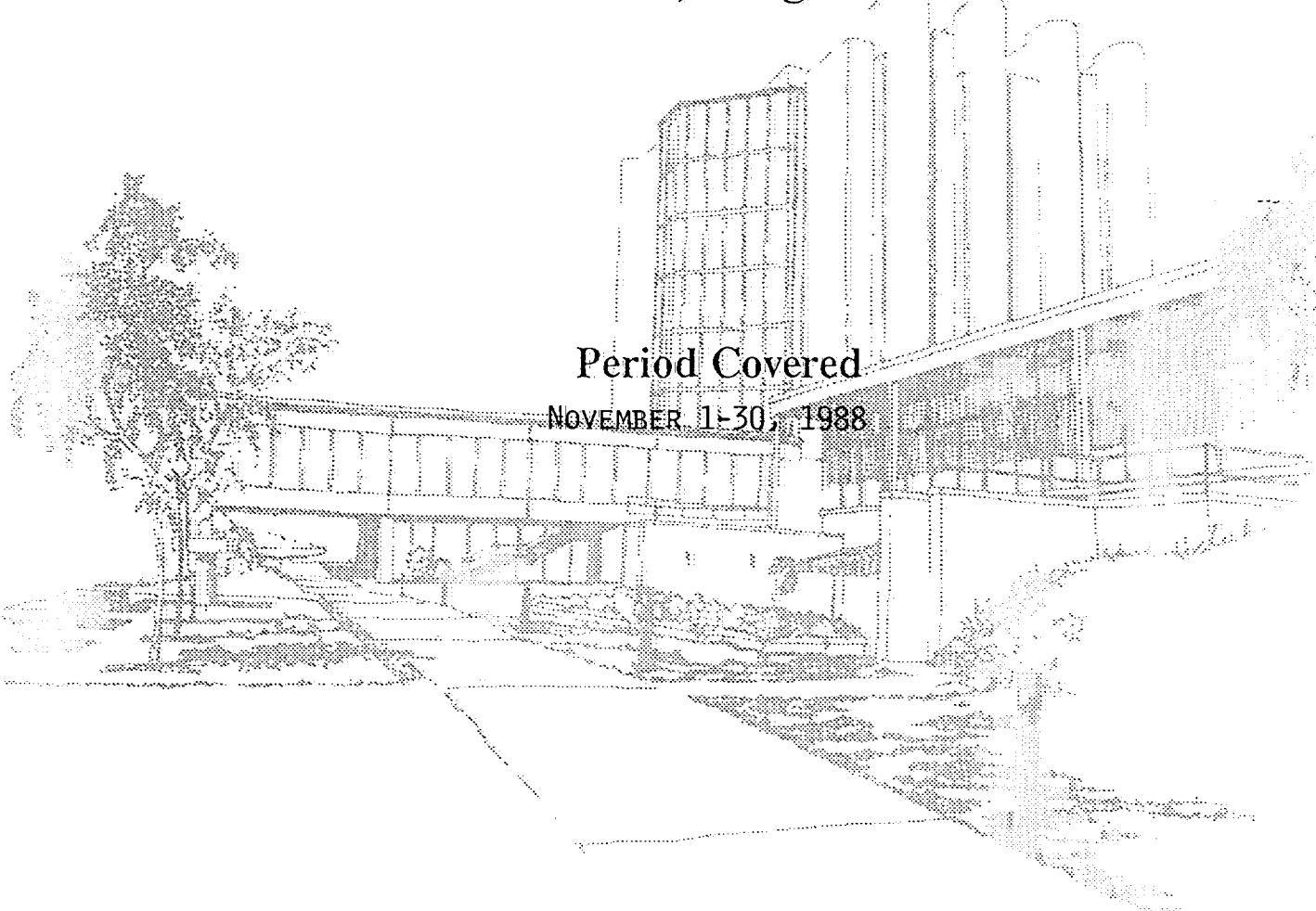


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PROJECT NUMBER: 2107  
PROJECT TITLE: Filter Research & Development  
PROJECT LEADER: C. J. Campbell  
PERIOD COVERED: November, 1988

#### I. FILTER RESEARCH & DEVELOPMENT GROUP

- A. Objective: Consolidate efforts on filter research and development into one group and actively pursue the development of improved filter systems.
- B. Status: Six models of Filtrona concentric filters, three core flow and three peripheral flow, have been analytically tested on a Merit Ultra Lights type cigarette. A higher RTD is required with a concentric filter in order to achieve the same tar delivery obtained from a conventional filter at an equivalent ventilation level. Subjective testing of a concentric model with a delivery equivalent to the Merit Ultra Light control is planned.

Samples of these six filters were sent to FTR for their testing. FTR has been considering using a 37.5mm peripheral flow concentric filter in a new brand test market. Samples supplied by Richmond were tested and deemed acceptable. FTR's initial specification for a 2 up, 75mm rod could be supplied by Filtrona in the time required for production startup. However, FTR's change to a 4 up, 150mm rod cannot be easily produced by Filtrona. A decision by FTR on this is pending.

Stackpole Carbon has been asked to develop an extruded carbon rod for possible use as a filter in place of loose carbon particles. Initial samples have been submitted and are now being prepared for evaluation. A meeting with Stackpole was held on 11/22 to examine the samples and to plan a course of action.

#### II. FILTER SUPPORT FOR MAJOR PROGRAMS

- A. Objective: Provide design assistance and potential new filter systems for major R&D programs.
- B. Status: Initial cursory tests with handmade samples evaluated by the Flavor Panel suggest that specialty filters may enhance subjectives of ART cigarettes. These filters consist of a peripheral flow concentric filter, a filter with potassium carbonate additive, and a filter with dipotassium phosphate additive. Machine made samples with additional ART blends will be evaluated.

Sample filter rods at 16.7mm circumference with a carbon on tow segment have been ordered for low sidestream Trim models. These are being produced by Filtrona in England and are due to be delivered the week of December 19.

2022221732

New formulations of PVA Menthol which use Benzoflex plasticizer have been tested and show a reduced ability to hold menthol when compared to the original PVA formula.

### III. CELLULOSE ACETATE WEB

- A. Objective: Explore the potential advantages of CA web for use as a high efficiency filter material.
- B. Status: Celanese has produced filters from CA web to our specifications. These are now on hand and will be fabricated by Semiworks into Merit Ultra Lights models for evaluation of performance advantages.
- C. Plans: Evaluate samples for efficiency and subjectives.

### IV. IMPROVED MENTHOL STABILITY

- A. Objective: Investigate methods of improving the stability of menthol delivery in smoke of aged cigarettes.
- B. Status: Preliminary results of an aging study of menthol cigarettes made from Filtrona heat set filter rods indicate that they maintain a more stable menthol in smoke delivery than Marlboro Lights Menthol control cigarettes. Heat set cigarettes delivered 65% of their original menthol after 28 days of accelerated ageing while the control Marlboro Lights delivered 47% of their original menthol. A follow up test is being planned to equalize initial menthol delivery, and to include a subjective comparison.

### V. TOW VENDOR R&D COORDINATION

- A. Objective: Coordinate specific requests for R&D work by Eastman and Celanese.
- B. Status: Merit Ultra Light cigarette models have been made with Eastman 1.6 dpf filters and are now in CI for analytical smoking. Similar models made with Filtrona UHF filters are now being made and will also be tested. These may allow the construction of ultra low delivery cigarettes with reduced ventilation. A more comprehensive test is being planned to include subjective evaluation.

An annual review of Celanese R&D efforts is set for December 7.

2022221733

PROJECT NUMBER: 2108  
 PROJECT TITLE: New Product Technology  
 PROJECT LEADER: W. T. Callaham  
 PERIOD COVERED: November, 1988

# I. PROJECT SAUNA

- A. Objective: Develop an acceptable product with a modified plastic fluted filter.
- B. Status: Cigarette models were prepared with several levels of front band dilution and cigarette paper dilution. These were evaluated by EEMA personnel and one prototype was subjectively preferred. This model had 80% laser rear band dilution, 12% ESP front band dilution, 130 Coresta ESP cigarette paper and minimal smoke blow back. The ISO delivery for this model and Saudi Barclay are as follows:

	<u>Sauna</u>		<u>Saudi Barclay</u>	
	<u>Open</u>	<u>Occluded</u>	<u>Open</u>	<u>Occluded</u>
ISO Tar, mg/cigt	0.6	11.3	1.5	11.8
ISO Nicotine, mg/cigt	0.14	0.94	0.18	0.96
Puff count, puffs/cigt	8.5	6.8	9.8	8.0

To further optimize the tar and nicotine deliveries for the Saudi market, a new blend was prepared with two different flavor systems. The previously preferred model was made with new fillers and cork-on-white tipping. Several of the cork-on-white tipplings investigated experienced activation of the hot melt precoating during laser perforation of the rear band making the tipping unuseable. Consequently, the rear band had to be perforated via ESP with an actual dilution of 70%. The ESP front band dilution was increased to 20% to better balance the dilution. Smoke blow back continued to be less than or equivalent to that of Saudi Barclay. The ISO deliveries for these models are as follows:

	<u>Old Blend</u>		<u>New Blend</u>		<u>New Blend</u>	
	<u>Old Flavor</u>		<u>Old Flavor</u>		<u>New Flavor</u>	
	<u>Open</u>	<u>Occl.</u>	<u>Open</u>	<u>Occl.</u>	<u>Open</u>	<u>Occl.</u>
ISO Tar, mg/cigt	2.6	11.4	3.0	10.6	2.5	10.2
ISO Nicotine, mg/cigt	0.27	0.95	0.30	0.81	0.25	0.80
Puff count, puffs/cigt	9.3	7.2	8.2	6.9	8.3	6.6

Internal subjective evaluation of these models indicated no preference for either flavor, therefore, the old flavor will continue to be used.

- C. Plans: Prototypes with the new blend and both flavor systems, will be remade with target dilution levels of 80% rear band (via laser) and both 12% and 20% front band (via ESP). These will be submitted to EEMA personnel for evaluation.

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## II. HUMIDOR PACK

- A. **Objective:** Develop a moisture release device for use in a cigarette pack which maintains the pack OV at a desired level.
- B. **Status:** Handmade packets were prepared with two different size Celgard "windows" and functionally tested versus the original packet design. After 25 days of laboratory and desert room ageing, the window packets are performing acceptably, with the packet having the largest window performing the better of the two. Pack OV results are as follows:

	Control No Packet	Humidor Pack		
		Standard Packet	Large Window	Small Window
Initial OV	13.8	13.8	13.8	13.8
Lab Conditions	13.5	14.7	14.2	14.1
Desert Room	8.7	11.6	11.5	11.1

Packets were also prepared with heated saturated solutions (no excess salt) and functionally tested. These performed acceptably, therefore, this change and the large Celgard window will be incorporated into the next packet production trial.

Reynolds Metals has supplied a modified EVA-foil laminate material for bonding with cellulose triacetate film (an alternate to Celgard). Packets are currently undergoing functional testing.

- C. **Plans:** A packet production trial will be conducted in mid to late December at Paco Pharmaceuticals. Packets from this trial will be used for functional testing and cigarette packer development trials. Also, a consumer test is being considered for late January, 1989 in the Middle East.

## III. KAYMICH MENTHOL APPLICATOR

- A. **Objective:** Evaluate a Kaymich menthol applicator as an alternative mentholation process.
- B. **Status:** Cigarette models from the most recent Kaymich mentholator trial have been tested analytically and subjectively against a menthol-on-foil control. Generally, the Kaymich models were subjectively variable in menthol response throughout three weeks of lab storage. The Kaymich models with the two higher menthol application levels (4.3 and 4.1 mg/cigt) were most comparable to the control after two weeks, however, the two lower level models (3.7 and 3.9 mg/cigt) did not deliver enough menthol in smoke. A puff-by-puff analysis of the two preferred Kaymich models versus the control after 17 days of storage indicated all three to be equivalent.

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- C. Plans: Another trial will be conducted to further study the subjective response of Kaymich menthol models as well as the menthol application uniformity. Spray-applied menthol and menthol-on-foil models will be used as controls. Additionally, mentholated filters will be investigated as a means of achieving a more uniform menthol delivery in a shorter period of time.

To assess the menthol uniformity during this trial, two approaches will be taken. First, sequential cigarettes will be collected and these will be analyzed for total menthol. Also, a high speed video recording will be made of the cigarette paper exiting the menthol nozzle.

#### IV. EMBOSSING TECHNOLOGY

- A. Objective: Explore embossing technology for potential new product development.
- B. Status: During debugging of the new laboratory embossing unit, several damaged components were found. These items are currently being corrected by the vendor.
- C. Plans: Continue providing support to Engineering as needed and pursue other applications for new product development.

#### V. MENTHOL-ON-FOIL

- A. Objective: Support the introduction of the menthol-on-foil process.
- B. Status: Mentholated foils were prepared for Product Development as requested.

The third mentholator control problem has not been corrected yet by Engineering due to priorities.

2022221736

PROJECT NUMBER: 2304  
PROJECT TITLE: Flavor Development - New Products  
PROJECT LEADER: Barbara G. Taylor  
PERIOD COVERED: November, 1988

I. LOW DENSITY ROD

- A. Objective: To achieve reduced tobacco weights in PM products via the Low Density process.
- B. Status: Filler for the Low Density rod process will be produced with 1x, 1.5x, and 2x levels of MF aftercut. Cigarettes will be made of each aftercut level using three filter designs to achieve the best analytical and subjective candidate to be compared to the Project Tomorrow candidate.

2022221737

PROJECT NUMBER: 2305  
PROJECT TITLE: Flavor Development-Brand Modification-Menthol/Distinctive  
PROJECT LEADER: H. M. Maxwell  
PERIOD COVERED: November, 1988

I. ALTERNATIVE FLAVORS:

- A. Objective: To identify and qualify alternative sources for flavoring materials.
- B. Status: Results of each replicate POL testing of alternative Jonex indicate no difference for each test. Results of the two POL's will be combined.

C. Plans:

Recommend Qualification                      December, 1988  
of the Alternative Jonex

II. PROJECT MOOG:

- A. Objective: To develop the expertise to produce cigarettes that are subjectively equivalent to Salem, Newport and Kool.
- B. Status: A K-type POL test was made in Semi-works, and was rejected due to analytical unacceptability. It will be remade. The S-type models were evaluated, and one was selected for further testing.

C. Plans:

K-type POL Remake                      December, 1988

III. MENTHOL RELEASE COMPOUND:

- A. Objective: To develop a mentholated charcoal filtered cigarette utilizing a menthol release compound. Also, to apply menthol release technology to other areas.
- B. Status: The Foam Bound Rod group is continuing to investigate problems of MGC application. Phase II clearance of the anethole/menthol compound has not been obtained yet.

C. Plans:

Cigarette Remake (MGC)                      To be scheduled

IV. ALTERNATE HUMECTANT PROGRAM:

- A. Objective: To develop the capability to produce cigarettes that are propylene glycol and glycerin-free, while maintaining product subjective integrity.

- B. Status: A series of models were made in small-scale to identify parameters required to achieve 16mg "tar." Analytical evaluations are in progress. Subjective evaluation(s) of 16mg cigarettes will be conducted when analyticals are complete.

V. PROJECT LEVO:

- A. Objective: To determine the subjective differences between natural and synthetic menthol, and identify techniques to lessen PM dependence on natural menthol.
- B. Status: Recent political developments indicate a need to pursue this program on a more urgent basis. Recent POL testing has given tentative indications regarding differences between natural and synthetic menthol. Additional testing is being initiated to verify these differences.

2022221739

PROJECT NUMBER: 2306  
PROJECT TITLE: Marlboro Standardization/International Support.  
PROJECT LEADER: Janet L. Spruill  
PERIOD COVERED: November, 1988.

I. MARLBORO STANDARDIZATION

- A. Objective: Analytical and subjective evaluations of production Marlboro KS/LS.
- B. Status: Standard Run VI completed at all factory locations with analytical and subjective evaluations underway.
- C. Plans: Evaluations of Standard Run VI.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL

- A. Objective: To provide subjective direction for programs within R&D and manufacturing locations.
- B. Status: Production runs of Alpine family under evaluations daily. Sixteen panels completed for the reporting period. Profiled 10 domestic brands.
- C. Plans: Provide assistance as needed.

III. PROJECT NATURAL

- A. Objective: To develop 85mm and 100mm full-flavored and lights prototypes using blend components and flavor systems which will result in a natural blended product.
- B. Status: POL 4447 (Natural Blend "C" Lights vs Marlboro Lights) released for testing. POL 3598 (Blend C using limited flavor ingredients vs Marlboro 85) has been made and submitted for analytical.
- C. Plans: Analytical and subjective evaluations of "Limited" model.

IV. FLAVOR RESOURCE DATA BANK

- A. Objective: Creation, customization and maintenance of flavor resource and data files for use by Flavor Development Division.
- B. Status: New vendor samples continue to be evaluated subjectively.
- C. Plans: Division updated with information.

V. INTERNATIONAL SUPPORT

2022221740

International Brands Smoking Panel

- A. Objective: Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.
- B. Status: Stockton Street start-up of PM Super Lights 83 FTB and Lark Milds 83 FTB were evaluated subjectively. Also, MC's start-up of Merit Lights-Japan were evaluated. Thirteen additional panels were completed during the reporting period. Eleven International brands were subjectively profiled.
- C. Plans: Provide assistance as needed.

PROJECT LIGHT/ULTRA

- A. Objective: Develop 7mg product for Japanese market.
- B. Status: Merit Lights for Japan began 11/14/88 at Manufacturing Center.
- C. Plans: Evaluate production samples.

PROJECT MOUNT ULTRA-JAPAN

- A. Objective: Explore the use of new blends, new flavor systems and different construction styles. Cigarettes will be in the low and ultra-low category for the Japanese market.
- B. Status: Based upon Danchi test results, further development work will be implemented.

PROJECT AVALON

- A. Objective: Development of casing and aftercut systems for a regular and mentholated product for the Asian market.
- B. Status: Evaluations of prototypes near completion.

PROJECT STARSHIP II

- A. Objective: Produce a 12mg cigarette for the Japanese market.
- B. Status: Program complete.

PROJECT OLYMPIC (KOREA)

- A. Objective: Development of a product to be competitive to Pine Tree King Size.
- B. Status: Casing and aftercut development in progress.

MARLBORO IMPROVEMENT PROGRAM (AUSTRALIA)

- A. Objective: Develop an Australian Marlboro subjectively closer to the U.S. Marlboro.
- B. Status: Blends A and C chosen fabricated in Australia.
- C. Plans: Evaluations of prototypes.

2022221742

PROJECT NUMBER: 2307  
PROJECT TITLE: Flavor Investigation/Processed Tobacco  
PROJECT LEADER: W. R. Raymond  
PERIOD COVERED: November, 1988

I. FLAVOR INVESTIGATION:

- A. Objective: To provide analytical support for activities related to development and application of flavoring materials.
- B. Results:
1. Analytical Support: Qualitative and quantitative A/C and casing composition data were provided for formula transmittals for Famous and Merit Lights (Japan). Casings and flavors were qualitatively and quantitatively analyzed for Marlboro Standardization Run #6 and for factory trials of Natural Marlboro Limited, Famous, Merit Lights (Japan) and Half-Pint. Analytical characterization continued of flavor systems for project ART. Semi-works monitoring and complete flavor analytical testing were conducted for POL 3609 cigarettes. Qualitative GC profile data and GCMS component identifications were provided for Hilton A/C.
  2. A collaborative study with R&D Engineering is in progress to optimize MF A/C spray application in Semi-works. Thus far, five of six planned large-scale A/C spray runs have been conducted, varying the rate of filler throughput. Flavor Development has provided analytical data for anethole content in A/C and on filler sampled at the A/C cylinder exit, from the final weigh belt and from the cut filler silo (78 samples total). The sixth run and associated analyses will be completed this week.
  3. Scale-up of Cooked Flavor Casing for ART to 25 gallon batch production has been completed in Semi-works. Specification data have been generated and plans are in progress to conduct 100-150 gallon production trials at the Flavor Center.

II. PROCESSED TOBACCO:

- A. Objective: To develop basic and applied knowledge for the purpose of improvement or selective modification of subjective properties of processed tobaccos.
- B. Results:

Control and test cigarettes for POL 3609 (testing utilization of post-ART stem in RL's) were remade, approved analytically and subjectively, and shipped November 17. The POL is scheduled to close out on December 16. At the substitution level being tested in RL's (1/6 of Bright stem), utilization of factory post-ART stem is projected to be ca. 50%. Evaluations continue to determine whether the remainder can be utilized in RCB.



PROJECT NUMBER: 4009  
PROJECT TITLE: Smoke Studies  
PROJECT LEADER: B. L. Goodman  
PERIOD COVERED: November, 1988

#### I. PROJECT STUDIO

- A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream visibility.
- B. Results: Cigarettes made in a 23 mm circumference configuration with a matrix of special  $\text{CaCO}_3$  papers from Kimberly-Clark were subjectively evaluated to determine the effect of different levels of MAP, CMC, and succinate. The papers had varying combinations of high and low levels of the three additives, based on estimates of what would be acceptable on a product. The level of MAP was either 0.4% or 1.6%, the CMC level 0.3% or 0.9%, and the succinate level 5% or 8%. Subjectively, the Studio panelists saw differences between the samples, with the low levels of additives being more acceptable. Sidestream visibilities were not significantly different for the high and the low levels. The reductions in visibility for the 23 mm circumference models were less than 40%. Additional models have been requested to see if double wrapping will increase the sidestream reduction and if a charcoal filter will improve the taste.

Double-wrapping was pursued on other models as well. An earlier version giving maximum sidestream visibility reduction (75%), which was achieved by using a perforated, high level of  $\text{Mg}(\text{OH})_2$  paper (35%) from Ecusta in combination with a slow burning, porous, thin innerliner from DeMauduit, gave a very high puff count. Additional models with faster burning  $\text{Mg}(\text{OH})_2$  papers were made with and without charcoal filters along with single wrap controls. These cigarettes are awaiting analytical data and subjective evaluations. Sidestream visibilities were in the 75% reduction range, but static burn times were still long. Another porous innerliner with a higher level of citrate has been received from DeMauduit for a potential increase in the burn rate without sacrificing sidestream reduction. DeMauduit is also making a thin paper with 10%  $\text{CaCO}_3$  as another faster burning alternative.

An innerliner sample from Kimberly-Clark has also been requested. Their version consists of an ultraporous tea bag paper made from woodpulp, laminated onto the outer wrap with a low level of CMC. The tea bag paper by itself has also been requested from K-C for direct comparison of the effect of the lamination process versus using two bobbins on our maker.

The search for subjectively better additives on the K-C paper was continued with Chemical Research personnel. In-house coatings of CMC with different substitutions were made, and other cellulose derivative coatings are in progress. Coating of tobacco solubles at a high concentration resulted in cigarettes that would not burn statically in the light extinction cell. Reduced concentrations will be tried next.

- C. **Plans:** Evaluate double wrap models at three circumferences for analytical, subjective and sidestream data.

Combine maximum sidestream visibility reduction from Ecusta's different  $Mg(OH)_2$  papers with the most desirable burn rate properties to design subjectively acceptable models.

Test charcoal filters as well as the double wrap concept on 17 mm circumference cigarettes. Evaluate Sol-Gel applications for sidestream reduction.

## II. TIPPING PAPER MODIFICATIONS

- A. **Objective:** Investigate modified tipping papers for flare-up, coal drop-off and paper composition.
- B. **Results:** The most recent samples from Ecusta of cork iron oxide tipping papers conforming to German regulations were tested in a Marlboro configuration. No flare-ups were observed. The coal drop-off rates were 17% and 63%. These papers meet the P.M. color standards, and will be used for a factory run by Operation Services. A small factory run will also be made with cork tipping paper from Malaucene. This paper gave 0% flare-up and only 1% coal drop-off. Qualification testing of this paper will be initiated.
- C. **Plans:** Obtain factory made samples for taste and odor tests, and repeat flare-up testing.

PROJECT NUMBER: 2304, 2305, 4015  
PROJECT TITLE: New Product Development  
PROJECT LEADER: B. G. Taylor, H. Maxwell, J. B. Easley, and L. S. Wu  
PERIOD COVERED: November, 1988

#### I. MARLBORO ULTRA LIGHTS

- A. Objective: To develop 85/100 mm Ultra Low (6 mg) candidates for Marlboro line extensions.
- B. Status:  
POL 4443 (MUL 85mm vs. Philip Morris Lights American 85mm) and POL 4442 (Half Pint 85mm vs. Philip Morris Lights American 85mm) have been cancelled.

The MC Panel smoked LDB-2R, LDB-3 and LDB-3 with modified flavors in the 85mm configuration. The results indicated no significant differences for acceptability, off-taste or added flavor. The LDB-3 at the .10 level of significance was lower in strength than the LDB-3 with modified flavors.

A factory trial was conducted at Stockton Street the week of 11/21/88 with 85mm and 100mm cigarettes produced in both cork-on-white and white tipping. All the cigarettes were made with the LDB-2 blend.

C. Plans:

Phase IV POL Tests - completed	1st Qtr., '89
Ad/Pack Testing	TBD
Test Market	2nd Qtr. '89

#### II. Trim I

- A. Objective: To develop ultraslim product candidates with 17mm circumference that demonstrate product advantages over Capri and Capri Menthol.
- B. Status: Based on internal evaluations, Trim Model 2, regular and menthol, was selected for POL 7182 (Regular vs. Capri) and POL 9088 (Menthol vs. Capri Menthol). POL cigarette production will be completed in December and the tests will be shipped after Christmas.

C. Plans:

POL Production (7182 and 9088)	Dec. '88
POL Shipment	Jan. '89
Test Market	TBD

III. Trim IV

- A. Objectives: To develop ultraslim product candidates with 17mm circumference with reduced visible sidestream that demonstrate product advantages over Capri and Capri Menthol.
- B. Status: Additional development work is underway to produce acceptable prototypes with respect to sidestream visibility reduction, subjective evaluations and analytical requirements. In addition to low sidestream paper evaluations, combined charcoal filters and doubled wrapped cigarette rods will be evaluated for this program.

C. Plans:

Double-Wrap Prototypes	Dec. '88
Combined Filter Prototypes	Jan. '89
Paper Evaluations	Ongoing
Test Market	TBD

IV. PROJECT NATURALA. Objective:

Phase I - To develop "All Natural" cigarette candidates with no additives to the tobacco.

- B. Status: Blend evaluations for spotting problem have been completed. Further work scheduled testing various paper modifications from Ecusta and Kimberly Clark. Cigarette specifications for PMI 100's Full Flavor Regular and Menthol have been written.

Flavored POL 3598 (97K Flavor system vs. Marlboro) was made week of 11/21/88. Awaiting C.I. data.

C. Plans:

Study Anti-Spotting Papers	on-going
Menthol	on-going

V. PROJECT EXTRA

- A. Objective: The development of 4-8 mg product candidates that have the taste of products with twice the tar.
- B. Status: The designs have been completed to test the theory of lower dilution due to lower rod RTD for low density products. The results indicated that for equivalent tar and puff count there is no dilution savings due to the higher availabilities in the low density configuration.

Filler for the Low Density rod process will be produced with 1x, 1.5x and 2x levels of MF aftercut. Cigarettes will be made with each aftercut level using three filter designs to achieve the best analytical and subjective candidate to be compared to the Project Tomorrow candidate.

A program to develop a 6mg free-standing cigarette which appeals to flavor low smokers was initiated. Three blends, recommended by the Leaf Department, have been evaluated with one chosen for Aftercut work with both CA and paper/CA filters. Four models have been made from which a candidate will be chosen to test on POL vs. Half Pint. This program has been named Project 605.

C. Plans:

Evaluate paper/ca filter variability	4th Qtr. '88
Evaluate alternate filters	On-going
Puff profile	On-going
Low density rod program	On-going

VI. Project 202

A. Objective: To develop a 2-3mg "Merit-type" cigarette for GCC.

B. Status: This is a spin-off of the Extra Project.

Modified Half Pint blend with 25% expanded, Half Pint casings and AC, paper/CA dual filter has been selected for the Saudi market. Specifications have been written.

C. Plans:

Specifications	11/88
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VII. Merit Super Lights

A. Objectives: To develop a 2mg "Merit-type" cigarette.

B. Status: A monadic non-menthol mapping POL has been requested for a new model (Model 2) that incorporates the 350°F expanded burley blend, modified Mount total blend casing and a chocolate/nutty-type aftercut. Model 2, the 25% BLDET model and PM Ultra Lights are being scheduled for MC panel smoking.

Aftercut 7695-178E is being subjectively evaluated on two blend models mentioned above. Both blends have been cased with Total blend casing 7695-163D.

C. Plans:

Model 2 POL test Production	1st Qtr. '89
Internal M.C. Panel Testing	1st Qtr. '89
Prototype Evaluations	Ongoing

VIII. PROJECT VALUE ENTRY

A. Objective: To develop products to compete in the Value Entry Categories.

B. Status:

Alpine

The Generic Alpine brand family has done well in Test Market and the plans are for National introduction early January 1989. Assisting with factory introduction of the new Generic Alpine brands and monitoring production startup for all six of the Alpine brands.

Target

POL 3608 (Target Model II 85mm vs Winston 85mm) was requested to determine the consumer acceptability of the new Target blend and flavor system versus Winston by Winston and other RJR smokers. The Target cigarettes have been analytically and subjectively approved. Currently awaiting the order of Winston cigarettes for the test.

C. Plans:

Alpine National Introduction	1/89
Target Ad/Pack Testing/Factory Trial	On-hold

XI. B&H KING SIZE

A. Objective: To develop a B&H 83mm box product with comparable subjectives of B&H 100 in both a full flavor and lights version, both nonmenthol and menthol.

B. Status: A series of Ad-Pack and Consumer Testings Studies have been requested by New York. The testing involves five different models: B&H blend - Regular and Menthol; and full flavor and lights; and Natural Blend C - Lights. All the packs have been shipped to New York.

Cigarettes are scheduled to be made the week of 12/12 using Natural Blend C in both lights and full flavor (regular and menthol).

C. Plans:

Blend and flavor work	On-going
Test Market	2nd Qtr. '89

X. Marlboro Blend Reformation

- A. Objectives: To remove off shore tobacco from Marlboro blend.
- B. Status: Phase XI cigarettes (Run I) were shipped to panellist in early November. The results of this test show favorable responses to blend 11 and 11-1. A repeat of Phase XI was produced at Stockton Street in early November but was rejected due to high tars. A remake is planned for early December.

C. Plans:

Phase XI repeat

12/88

PROJECT NUMBER: 4022  
PROJECT TITLE: International Product Development  
PERIOD COVERED: November, 1988  
WRITTEN BY: A. H. Confer

I. PROJECT DRAGON (PEOPLES REPUBLIC OF CHINA)

- A. Objective: Develop a "blended" KS cigarette to be a non-PM trademark owned and manufactured by CNTC. Tar targets: 17-19 mg (GCF-2 factory), and less than 15 mg (ZCF factory).
- B. Results: A Chinese delegation of 8 were entertained in Richmond November 15-18. Prototypes from the GCF-2 factory revealed the 20% off-shore inclusion blend was preferred to the 100% local blend, but it is still not satisfactory.
- C. Plans: More flavors will be prepared and shipped to each factory next month to make prototypes using two blends co-designed by C. Brumberg and the Chinese.

II. PROJECT TANG (INDONESIA)

- A. Objective: Develop subjectively and analytically identical product to convert locally manufactured MF LS from cut filler to BBS without expanded components.
- B. Results: First generation prototypes have been designed using two Leaf Department blends.
- C. Plans: Primary and Make-pack runs are scheduled in the R&D Semiworks for the first half of December. A visit to the Bentoel factory to assess their primary capabilities is planned for early January.

III. PROJECT PAGE 1 AND 2 (TAIWAN)

- A. Objective: Develop an American blended export product in a full flavor and lights version for a potentially emerging price/value market segment.
- B. Results: Full flavor and lights prototypes have been developed using two different blends. They were smoked analytically and subjectively in Richmond.
- C. Plans: Let Hong Kong management smoke the prototypes and render a decision.



IV. Project Bella (Hong Kong)

- A. Objective: Develop a lights box line extension of the parent Virginia Slims menthol cigarette.
- B. Results: Mentholated pack foil and cigarettes were produced in Louisville using three levels of menthol (50, 60, and 70 mg per pack).
- C. Plans: Evaluate the prototypes locally, and if found acceptable, send them to Hong Kong to select a candidate to launch.

PROJECT NUMBER: 4024  
PROJECT TITLE: Japan Product Development  
PERIOD COVERED: November, 1988  
WRITTEN BY: J. N. Smith

I. MERIT LIGHTS

- A. Objective: Development of a 7 mg tar product to compete with Mild Seven Lights among mainstream Japanese smokers.
- B. Status: A factory trial of Merit Lights KS SP was conducted on November 8 at the Manufacturing Center to finalize cigarette specifications. Initial production of the product began on November 14. It is planned to build an inventory of 775 million cigarettes for a national introduction in February 1989.
- C. Plans: Nationwide Consumer Testing Institute (a branch of U.S. Testing) will sample, test, and certify the FTC tar deliveries of Merit Lights KS SP and Mild Seven Lights 80 mm Soft Pack to support PMKK's advertising claims.

II. MARLBORO

- A. Objective: Improvement to the subjective response of the Marlboro Full Flavor product.
- B. Status: Specification packages for both the new Marlboro King Size and 100's Soft Pack were sent to PM Asia for review.
- C. Plans: Discussions with JTI are planned for December.

III. MOMENT

- A. Objective: Development of a menthol product acceptable to the Japanese consumer.
- B. Status: The second Danchi test of the Moment series was shipped on November 4. The third test in this series was rejected due to high menthol in smoke deliveries on two of the models.
- C. Plans: The third Danchi test will be remade in Semiworks to the target menthol deliveries.

PROJECT NUMBER: 5001  
PROJECT TITLE: Packaging Studies  
PROJECT LEADER: H. R. Dunaway  
PERIOD COVERED: November, 1988

#### PACKAGING STUDIES

A. Objective: Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. Status/Plans:

Residual Printing Solvents in Packaging Materials: Evaluation of the following items was coordinated in the past month.

- B&H FF Men KS FT 20 for Ad-Pack (Alford)
- Marlboro Cork Tipping w/Toluol Free Ink (Ecusta)
- Project C-2 FT 20 (Alford)
- Merit "Heavy Up" KS&100 Pet-G 20/100 (Klearfold)
- Indonesian Marl LS FT 20 made with both Tako and Ivory boards
- Merit "Heavy-Up" promotional postcard
- Offset Carton Insert with 100% ink and varnish coverage
- DJW-2042 Water Base/urethane Lacquer
- Chemical 10 Antifoam
- RJC6011 Toluol Free Solvent Lacquer
- BYK-052 Defoamer
- JCCCK Fluo & 16-2300 Videojet Invisible UV inks  
(To print vendor material tracking information inside cartons.)

Poly Laminated Blanks and Cartons: Samples were packaged at the Semi-Works using six combinations of test and control materials. The 12 week OV study will begin early December to evaluate moisture retention.

Subjective Evaluation of Packaging Materials: Samples were prepared, aged, and submitted for the subjective smoking evaluation of the following packaging materials:

- Teich 15# Silver Foil (New Supplier)
- Alcan 15# Silver Foil with new Tissue Paper Source
- National 34-2928 Hotmelt for use on Promo Pet-G 1/2 Cartons.
- KTI Silver Line Tear Tape with Pressure Sensitive Adhesive
- Klearfold Pet-G 5-Pack Promotional Cartons

Project Fox: Cigarettes packed in the latest plastic FT 20 made with Philips 55-500 HD Polyethylene and CPLPE green pigment, were rejected by the Flavor Development smoking panel. Cigarettes packed in the box samples after conditioning at 135° F for 8 and 24 hours, to drive off volatiles, were found comparable to controls with the 24 hour version preferred.

Fiber-Lam, Inc. Shipping Cases: A 12 week storage study is underway to evaluate moisture protection provided by cases with a polyethylene

coating within the laminated liner board. After six weeks storage under Desert conditions, product packed in the test case has lost .3% OV while that packed in the conventional case has lost 2.7% OV.

Miscellaneous: A double-sided tape from a new source is under evaluation, for NY Marketing Services, to replace the material currently used on POS promotions.

PROJECT NUMBER: 0400  
PROJECT TITLE: Low Density Rod Development  
PROJECT LEADER: R. S. Mullins  
PERIOD COVERED: November 1988

I. LOW DENSITY ROD

- A. Objective: Develop a continuous process for the production of reduced density cigarettes.
- B. Results: Analytical testing of the low density cigarette models produced to determine if reduced cigarette density allows a significant reduction in dilution, at constant delivery, puff count, and RTD has been completed. Analysis of the results by New Products is in progress.

Production of the second set of samples (Marlboro configuration) for Project Tomorrow has been completed. The samples are currently being weight selected prior to packing and testing. Following analytical testing and subjective screening by an expert panel, the cigarettes will be subjectively evaluated by the M/C panel.

Production of samples for the second aftercut tracking study was also completed. However, because of the sample load generated by the Semiworks aftercut work and by the ART program, Flavor Development cannot analyze the samples collected in the study for anethole content. The bottleneck in conducting the analyses is the extraction step, which requires 2 hours per sample. Flavor Development has agreed to train one of our technicians in the extraction process and to analyze the extracted samples.

No significant problems were encountered in ripping bound cigarettes in a test conducted at Stockton Street. The filler yield for the bound cigarettes was 67% while similar unbound cigarettes had a yield of 71%. No problems were encountered with either filler or binder from the bound cigarettes building up in the equipment.

Low density cigarettes were made with wet (15.7% OV) and dry (13.5% OV) batch-coated filler in a preliminary test of the effect of starting OV on cigarette quality. Visually, the wetter filler appeared to produce a better bound rod and to generate less dust and shorts. Sample cigarettes have been submitted to MEL for firmness and loose ends testing. Additional testing is planned to further investigate the effect of starting OV and to determine whether any effect might be attributable to the movement of moisture from the tobacco through the pectin film at higher OVs.

- C. Plans: Complete the analysis of the samples from the second aftercut study. Design and conduct a test to evaluate the effect of initial filler OV on the firmness of low density cigarettes. Evaluate moisture application to the filler in the chimney using an ultrasonic nozzle. Produce samples having a 40% density reduction for puff x puff TPM analysis to determine if this reduction is sufficient to significantly alter the vapor/particulate ratio of the smoke.

## II. Kaymich Menthol Applicator

- A. Objective: Evaluate the Kaymich direct menthol applicator as an alternative mentholation process.
- B. Results: Rheological studies on both natural and synthetic menthol indicated that crystallization did not begin to occur until the temperature was reduced to 36° C, well below the published freezing point of 42° C. These studies were carried out at shear rates from 0 to 500 inverse seconds, which approximate the shear rates expected in the temperature controlled flow lines and nozzle of the Kaymich unit.

An outside lab, Springborn Laboratories, which can perform thermal analysis of menthol has been identified. Samples of both natural and synthetic menthol have been sent to the lab for determination of transition temperature, heat of fusion, and  $C_p$ .

Two nozzles designed to apply menthol directly to the tobacco stream have been received from Kaymich. One nozzle protrudes through the side rail to apply molten menthol to the side of the tobacco stream while the other nozzle applies the menthol to the bottom of the tobacco stream using air atomization. A spare side rail has been obtained from the MC and will be modified as needed to accommodate these nozzles.

The Louisville factory was visited to gain an understanding of the production concerns associated with the current menthol-on-foil application process and to identify potential problems with implementing the Kaymich process. A meeting was also held with Operations Services personnel to discuss manufacturing considerations associated with the Kaymich process.

- C. Plans: Assist New Products in producing samples for an aging study to compare the analytical and subjective performance of cigarettes produced via the Kaymich and menthol-on-foil processes. Install and evaluate the nozzles designed to apply menthol directly to the tobacco stream.

PROJECT NUMBER: 1307  
PROJECT TITLE: Reconstituted Tobacco Development  
PROJECT LEADER: R. G. Uhl  
PERIOD COVERED: November, 1988

## I. IMPROVED SHEET PROPERTIES

A. Objective: Improve the physical characteristics and blend properties of reconstituted sheet materials.

B. Results:

1. ART Project - POL cigarettes to evaluate the acceptability of using half of the ART stems in pilot RL were mailed to panelists. Results should be available in January.

The stems from six ART runs (236-9, 241-2) in which the ART filler was precased with PG/glycerine were blended together and incorporated in pilot RL at both the half and full utilization levels. These are being made into small-scale blended cigarettes for subjective screening. The control and full utilization sheets are available in POL quantities if precasing represents a subjective improvement warranting further testing.

2. Humectants - Glycerine-free RCB handsheets were made with several levels of supplemental sugars (isosweet and sucrose) to determine if this would improve the subjective properties. The subjective shortfall in previous test products appears more related to the absence of class tobacco casings when CT feedstock is replaced by stemmery scrap to produce a glycerine-free RCB, than it is to the removal of glycerine. These sheets will be screened in handmade 100% cigarettes.

Semiworks survivability testing was completed on pilot RL sheets containing individual humectants (4% glycerine, 4% PG, 4% isosweet, or no humectant). Combined longs plus mediums exit the maker garniture showed no effect due to cutter OV, but had a strong direct relationship to maker OV. Piece size versus maker OV plots for three of the sheets (glycerine, isosweet, no humectant) were superimposed, indicating that moisture content alone governs RL survivability. The PG sheet showed lower survivability on an equal OV basis; correcting OV results for PG volatility in the OV test gave the PG sheet at least equal survivability on a constant moisture basis.

C. Plans:

1. Provide RL pilot plant support for the POL evaluation of ART stems in RL.
2. Expedite subjective evaluation of ART stems in RCB handsheets.

3. Expedite subjective evaluation of glycerine-free RCB handsheets made with additional sugars.

## II. SUBJECTIVE MODIFICATION OF RL

A. Objective: Improve or modify the subjective character of RL.

B. Results:

1. Dry Flavor Replacement - Flavor Development is continuing the analytical evaluation of vendor native extracts of liquid flavors following vendor modifications of the roasting process. These flavors will be evaluated in pilot RL when they become available.
2. Modified 150B - The Branson ultrasound processing unit was installed and tested. Size press nip rejection trials with Modified 150B size were initiated.

C. Plans:

1. Produce pilot RL to evaluate liquid flavors as available.
2. Complete trials to determine if ultrasound will break up Modified 150B size precipitates.

## III. CIGARETTE PAPER DEVELOPMENT

A. Objective: Support development of proprietary low sidestream cigarette papers.

B. Results:

1. Handsheets - Cigarette paper handsheets were produced to evaluate experimental filler materials including magnesium carbonate, dolomite (a natural mineral combining calcium and magnesium carbonates), the Pfizer Multiflex MM fine (0.07 micron) calcium carbonate used at Maine, and Pfizer experimental Omega Fine (0.02 micron) calcium carbonate. The new laboratory size press was used to successfully size these handsheets and paper from the September University of Maine trials with potassium succinate (10% add-on).

Experimental bilayer handsheets were also produced. Each layer was made at 25 gm/m<sup>2</sup> (half the normal sheet weight) and contained a different filler. The two layers were then pressure laminated using the hydraulic flat press.

2. Pilot Trials - A second set of trials was conducted on the University of Maine pilot paper machine. The absence of beater power load monitoring equipment made it difficult to reproduce the same degree of flax stock refining that gave the best results in September; Maine is addressing this problem.



and is also improving the mechanism for sheet pickup off of the paper machine wire. The Claflin refiner was successfully used for final freeness adjustment of the beaten stock; this required the use of a stock screen to remove fiber knots formed in the refiner. Moving the stock screen to immediately before the paper machine headbox (knot removal at the last possible moment) improved the appearance and runnability of the paper.

The new dandy roll did not produce sharp CD (cross directional) watermark lines; an etched roll installed in a third wet press position will be required for this purpose. The plain dandy was reinstalled for the remaining trials.

Subsequent beater adjustments resulted in the production of papers with Coresta porosities of 7-8 at a sheet weight of 40 gm/m<sup>2</sup> and containing 30% calcium carbonate (Pfizer Multiflex MM). Subsequent mechanical failure of the second press precluded production of significant bobbin quantities.

Maine is constructing and installing a secondary headbox to evaluate the feasibility of producing bilayer sheets. Trials for this purpose are scheduled for December. These trials will also evaluate the other equipment modifications made by Maine, and will allow the production of additional bobbins of low porosity paper for testing at R&D.

C. Plans:

1. Evaluate alternate (non-flax) fiber types as a means of improving cigarette paper properties.
2. Conduct a third set of trials at the University of Maine to evaluate the feasibility of producing bilayer papers.

PROJECT NUMBER: 1333  
PROJECT TITLE: Semiworks Process Control  
PROJECT LEADER: D. A. Phan  
PERIOD COVERED: November, 1988

A. Objective: Evaluate and revise the process control and data acquisition system to improve processing performance and production quality.

B. Results:

Hauni HT Steam Tunnel Installation (Oliver) - Continue to support Dr. Cho's program to evaluate the Hauni steam tunnel. Modifications to the weighbelt at the inlet of the tunnel will be performed the week of December 5 to increase its speed and reduce tobacco bed depth. This is to provide better tobacco feed to the tunnel. Other instruments were installed to monitor the spray steam temperature and pressure, and tobacco temperature at the discharge of the tunnel and the inlet of the Adt dryer.

Temperature Control System for Scandia Overwrappers (Osmalov/Phan/Sims) Additional mechanical modifications to one Scandia at the Stockton St. factory were completed. Initial results indicate that significant improvement in pack-seal quality has been obtained. Further evaluation is being conducted by factory personnel.

Second Aftercut Flavor Cylinder (Phan/Sims) - Electrical and control installation for the flavor system and associated conveyors were completed and checked out. The system is ready to accommodate further flavor application improvement being conducted by the Semiworks Process Development group.

Vacuum Conditioner Data Highway Connection (Sims) - Connection of the Provox data highway to the stand-alone Provox system at the vacuum conditioner panel was completed and checked out. This will allow better maintenance of conditioner control software from the central system, and opportunity of trending some important process variables at that location.

Automatic Damper System for The Adt Dryer (Sims/Medek) - Work is underway to provide an automatic system for remote switching of the inlet and exhaust air dampers from the control room for co-current or counter-current air flow modes. Automatic dampers were installed and checked out. Other solenoid valves for the hood pressure control loop will be installed and checked out the week of December 12.

C. Plans:

Complete as-built documentation for the second aftercut flavor system. Complete the remaining items for the Adt dryer automatic damper system, and the Hauni steam tunnel infeed weighbelt. Continue the development of the Onspec control software and qualification of the Computrac moisture analyzer. Continue providing electrical plant engineering support to the Semiworks and conduct routine QA functions.

PROJECT NUMBER: 1503  
PROJECT TITLE: Modified Smoking Materials  
PROJECT LEADER: W. A. Nichols  
PERIOD COVERED: November, 1988

I. LOW DENSITY ROD - BINDER APPLICATION

- A. Objective: Produce precoated filler via the batch or continuous process to support the current product and process development efforts on the low density rod program. Develop a method of pre-applying binders to the tobacco in a manner that can be scaled up to commercial scale.
- B. Results: To minimize tobacco build-up in the coating cylinder, the continuous process requires the filler be passed twice through the cylinder adding 3% pectin in each pass. Two tests were conducted at increased solution concentrations to permit the total add-on in one pass. While processing through the cylinder was feasible, resulting cigarette rod firmness of the low density rods was inadequate. These low firmness values may have been due to low filler OV (11.7%) at the maker or non-uniform pectin coating. Testing using higher OV filler is planned.

To evaluate binders as potential filler bonding agents, a binder reactivation device (1 g samples) is being assembled. This unit will be used to attain preliminary processing requirements of various binders and to evaluate reactivation of samples from alternate binder application methods.

920 lbs. of Marlboro filler for Project Tomorrow samples was coated using the continuous process. All lamina filler at various OVs is being produced to evaluate the optimum OV for cigarette fabrication.

- C. Plans: Tracer investigations will be completed and standardized testing established. Costs and specifications on a scaled-up continuous coating reel will be discussed with FMC, the supplier of the existing unit.

PROJECT NUMBER: 1806  
PROJECT TITLE: New Tobacco Processes  
SECTION LEADER: S. R. Wagoner  
PERIOD COVERED: November, 1988

I. PROJECT ART - PILOT PLANT SUPPORT

- A. Objective: To provide processes for converting and casing stem materials for the Bermuda Hundred Pilot Plant.
- B. Results: Batches of Louisville CRS (cut at 150 cpi) were cased with monopotassium citrate and shipped to the Bermuda Hundred Pilot Plant on a daily basis. For designated runs and Product Development purposes, PG was included in the casing solution.
- C. Plans: Continue to produce stem products as required by Project ART.

II. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

- A. Objective: To conduct trials providing information for development of the ART commercial process.
- B. Results: With the support of Quality Audit and Quality Engineering, cigarette inspections for critical spots continued on extracted ART filler. Levels higher than normal PM production were found for all typically processed ART fillers (10-50 critical spots/2000 cig vs 1-2 for normal production). These levels appear to vary depending upon the humectant type and level applied to the pre-ART filler. Further inspections will be conducted for verification. Cigarettes made from 100% expanded ART filler also yielded higher than normal spotting levels.

ART runs 251/252 were steam conditioned in the Hauni HT steam tunnel to determine any effects on subjective, chemical, or spotting analyses. QA and QE cigarette inspections yielded 3.0 and 4.8 critical spots/2000 cig, respectively. These are the lowest values to date for extracted filler. Since these cigarettes contained no expanded material or aftercut flavor, another trial will be conducted, and cigarettes made from finished filler will be analyzed.

In an effort to scale-up previous laboratory experimentation that showed decreased spotting levels from ART filler that was dried in a vacuum oven, ART filler was treated in the Semiworks vacuum conditioner with a modified bright/DIET cycle. QE inspection did not show the reduction in spots observed in the preliminary tests. However, there was a reduction in ammonia content of approximately 0.1%. Trials will be repeated.

Expansion runs of ART filler at a tower temperature of 500F were carried out for Product Development work. In addition, the MC DIET system is being examined for potential sources of

contamination when expanding ART filler. Analysis of the CO<sub>2</sub> showed a nicotine level of 2 ppm which should not add to the level of the filler. Analysis of the tower gas will be conducted, and a short test run (1000 lb) with ART filler is being planned.

- C. **Plans:** Continue to quantify the level of cigarette spotting caused by ART filler. Conduct further post-ART tobacco treatment tests with the Hauni HT tunnel and the vacuum conditioner to determine the effect on spotting.

Complete investigation of the MC DIET system to assure that ART filler can be expanded without contamination.

In coordination with the Semiworks and Product Development, conduct commercial simulation trials for the purpose of setting primary processing specifications for the commercial facility.

### III. BINDER DEVELOPMENT

- A. **Objective:** Develop methods to produce binder systems for the foam bonded ends and low density rod programs.
- B. **Results:** For the foam bonded ends program, samples of 5% NaCMC with 0.5, 0.75, and 1.0% licorice were prepared for foam tests and machinability trials. A licorice content of 0.75% appears to be the lower limit to achieve a stable foam. Foam breakdown times, which need to be minimized from a processing standpoint, were found to be in the range of 40-60 seconds. Yucca will be tested as an alternative foaming agent to licorice.

For the reduced density rod program, contact angle measurements of pectin solutions containing 2-8% solids were conducted to determine the degree of penetration. Analysis on RL was completed, and testing with bright, burley, and Oriental strips is continuing.

- C. **Plans:** Continue to provide support as required to the above programs.

### IV. TMCI-ASTA SHEET

- A. **Objective:** To develop a subjectively and physically acceptable reconstituted tobacco sheet using the TMCI process and PM-RCB technology for international application.
- B. **Results:** Tarragona plant modifications were completed in November and TMCI started plant evaluation trials using the Microflake process. Preparations were made to run ASTA trials if these modifications prove to be successful. Samples of ASTA product from the May 1988 trials were sent to TSA for analytical comparison with PM results before these ASTA trials begin. Review of the drawings for the Cadiz installation continued, and

sealant and gasket materials approved by PM were sent to TSA for use in Cadiz.

Trials were run by European R&D in the FTR Monique process using finely ground tobacco dust, which greatly improved their sheet quality. It was also possible to run a higher solids content of the casting slurry which reduces the drying load of this sheet casting process. The details of these trials will be reported by FTR.

C. Plans:

Evaluate the mechanical modifications to improve the Tarragona plant operation by running trials on 12/13/88 using tobacco ground in Nuway to 95% <400 mesh).

Witness one week of satisfactory ASTA process operation using the normal tobacco grind before running trials to optimize tobacco particle size in Tarragona in January 1989.

PROJECT NUMBER: 1810  
PROJECT TITLE: Project ART  
PROJECT LEADER: Ravi Prasad  
PERIOD COVERED: November, 1988

I. PROJECT ART

- A. Objective: To support commercial Plant Design and Flavor Development objectives at the Bermuda Hundred Pilot Plant.
- B. Results: Priority at the Pilot Plant continued to be in support of the Product Development work. Process conditions for flavor development remained fixed as follows:

Extraction Pressure	3800 psi
Extraction Temperature	140°F
Filler Ordering	3% AB, 26% OV
Stem Precasing	12% K-citrate, 35% OV
M/M (lbs CO <sub>2</sub> /lb tobacco)	200

After extraction, the filler was "fluffed" at the Pilot Plant to remove any impregnation. This allowed successful downstream processing for Flavor Development activity, e.g., wet casing prior to drying and aftercut.

Addition of humectants (PG and glycerin) was implemented at the Pilot Plant to provide subjective benefits. A series of runs at various levels of PG and glycerin was conducted to determine the best flavor/process option. Subjective evaluations are in progress.

In support of the commercial plant design, two prototype on-line nicotine monitors were ordered for evaluation. Current schedule calls for delivery and installation of the first unit in January 1989.

To finalize the nicotine scrubber design of the commercial plant, nicotine laden carbon was thermally treated at the pilot plant. The carbon temperature reached the target of 400°F, with a required heat up time of 10 hours. Cool down required 12 hours. These times are long but acceptable for commercial operation.

A corrosion inspection program is being instituted at the Pilot Plant. Plans are to complete 100% ultrasonic inspection of high pressure pipe welds by the 1st quarter of 1989. In addition a combined ultrasonic and dye-penetrant inspection of the pressure vessels is being planned.

- C. Plans: Continue production in support of Flavor Development activity.

PROJECT NUMBER: 1101  
PROJECT TITLE: Entomological Research  
PROJECT LEADER: Dr. L. Faustini  
WRITTEN BY: L. Ryan  
PERIOD COVERED: November, 1988

I. PHYSIOLOGICAL STUDIES OF THE CIGARETTE BEETLE (CB)

A. METHOPRENE

1. Objective: To determine the most efficient use of the insect growth regulator (methoprene) within R&D's CB management program.
2. Results: There appears to be no sufficient loss of methoprene during the expansion process (1).
  - Studies are underway to determine the optimal methoprene dose for stems and scraps (2).
  - Discussions with Analytical personnel (3) regarding methods development to detect methoprene residues below 2 ppm and on Oriental tobacco were reported (4).

B. GLOBAL ECOLOGY

1. Objective: To determine the distribution and abundance of the CB as it relates to the processing of tobacco into cigarettes.
2. Results: Program initiated in Burley extruders at Bowling Green and Lexington and the Burley stemmery at Lexington. All Processing facilities belong to Southwestern Tobacco Company itself a Universal Leaf Company (5).

C. PESTICIDES

1. Objective: Provide a list of pesticides acceptable for potential use at PM facilities.
2. Results: Various consultations on pesticides were documented this month (6-10).

D. SERVICE TO OTHERS

1. Objective: To provide technical services to areas outside R&D.
2. Results: Recommendations for handling bulk RL, BL, and OR to prevent CB infestations were given (11).
  - Advice on implementing 100% Kabat® usage was given at a meeting held to address this topic (12).
  - Methoprene residues on Colombian strip were determined (8).



**E. OTHER**

- L. Ryan attended an "Advanced Course on Pest Recognition" (13).
- Polyethylene lined scrap tobacco, phosphine fumigated at Park 500 was cleared for use (14).
- D. L. Faustini assisted Miller Brewing Company, Fort Worth, TX with the carbon dioxide fumigation of an empty grain silo.
- D. L. Faustini attended an AMA "Basic Project Management Course" (15).

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3. Ryan, L. Memo to Distribution. Methoprene Residues Detection. 10/3/88.
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5. Ryan, L. Memo to Faustini, D. L. Cigarette Beetle (CB) Pheromone trap monitoring program at Universal Leaf Tobacco Company, Inc. (ULT) Facilities. 11/8/88.
6. Ryan, L. Memo to Schafer, D. Pesticides used in Public Warehouses. 11/14/88.
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9. Faustini, D. L. Memo to Ellis, C. K. General Foods Conversation. 11/1/88.
10. Faustini, D. L. Memo to Schulthess, D. Contact Space Spray Insecticides. 10/14/88,
11. Faustini, D. L. Memo to Haller, D. Bulk Tobacco handling process - Cigarette Beetle Concerns. 11/10/88.
12. Koval, G. A. Memo to Distribution. Plan for 100% Kabat® usage. 10/24/88.
13. Ryan, L. Memo to Faustini, D. L. Okumura Biological Institute (OBI) Course. 11/11/88.
14. Lehman, R. Notebook #8740 p. 1.

15. Faustini, D. L. Memo to Distribution. Trip Report - Basic Project Management Course, AMA, Wash. D.C. 10/13/88.

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PROJECT NUMBER: 1620  
PROJECT TITLE: Electrophysiological Studies  
PROJECT LEADER: F. P. Gullotta  
WRITTEN BY: C. S. Hayes  
PERIOD COVERED: November, 1988

# I. NASAL EVENT-RELATED POTENTIALS (NERPs)

A. Objective: To develop methods to objectively and reliably evaluate human responses to cigarettes, smoke constituents and tobacco flavorants.

## B. Results:

### Cognitive NERP Study

Cognitive NERP experiments comparing a mixture of natural and synthetic menthol to 100% synthetic menthol (MM/SM) were completed in ten subjects utilizing the cognitive odd-ball paradigm.<sup>1</sup> Both the electrophysiological and the psychophysical data demonstrate that subjects are able to differentiate between the mixture and the synthetic menthol. A late positive component (LPC) was recorded to the mixture targets that was not apparent or greatly diminished to the synthetic standards.

Thus far, all ten subjects have been tested under three menthol discrimination conditions: 100% natural menthol vs CO<sub>2</sub> (NM/CO<sub>2</sub>), 100% natural vs 100% synthetic menthol (NM/SM) and the synthetic/natural mixture vs 100% synthetic menthol comparison (MM/SM). Statistical analyses reveal that for each of the discriminations, the NERPs to the targets are significantly different ( $p < .001$ ) from the NERPs to the standard stimuli with respect to the late positive component (LPC). That is, the target NERPs contain a LPC that is not present or greatly diminished in the standard NERPs. As expected, the data reveal that the latter two menthol discriminations were more difficult than the NM/CO<sub>2</sub> discrimination. Stimulus reaction times were longer and the LPCs were longer in latency to the NM/SM and MM/SM discriminations as compared to the NM/CO<sub>2</sub> condition. Also, the LPCs to the NM/SM and MM/SM discriminations were smaller in amplitude and overall area compared to the NM/CO<sub>2</sub> LPCs. For most subjects, the MM/SM discrimination was slightly more difficult than the NM/SM condition. Reaction times were slightly longer, and the LPC measurements were in the appropriate direction to indicate a more difficult discrimination.

These data clearly demonstrate the utility of the cognitive NERP technique for providing an index of discriminability for menthol and other flavorants. The technique can be employed not only to determine whether subjects can discriminate among various flavorants, but also the degree to which they are discriminating. Statistical analyses are currently being conducted comparing hits (correctly responding to the target stimulus), misses (failure to identify the target stimulus), correct rejections (correct

identification of the standard stimulus as a non-target) and false alarms (responding to a standard stimulus as if it were a target). These analyses should provide additional insight into the discrimination process.

- C. **Plans:** Continue statistical analyses of the electrophysiological (NERP) and psychophysical data from each of the three menthol discriminations. Following the completion of modifications to the olfactometer, plans are to retest some subjects in the NM/SM comparison switching the target and the standard stimuli. Other plans include varying the ratio of natural to synthetic menthol in the mixture for comparison with synthetic menthol.

D. **References:**

1. Martin, B. R. Notebook No. 8689, pp. 110-159.

PROJECT NUMBER: 1704  
PROJECT TITLE: Supercritical Fluid Processes  
PROJECT LEADER: T. M. Howell  
PERIOD COVERED: November 1988

#### I. LOW NICOTINE

A. Objective: Understand solubility behavior of tobacco wax solubles in SC-CO<sub>2</sub>.

B. Results: Last month it was reported that tobacco solubles are not saturated in SC-CO<sub>2</sub> at process conditions but begin to precipitate at about 3000 psig as the pressure is reduced at constant temperature. Additional work has shown that OV in the range of 8% to 25% does not influence the cloud point.

Plans are to evaluate the effects of the water on non-uniform deposition of the waxes. That is, the waxes precipitate prior to reaching the dew point of the water so it is not known how water precipitation affects the growth and suspension of the tobacco solubles.

C. Plans: Continue developing this technique in order to better understand wax deposition and spotting during our current let-down procedures.

#### II. LOW NICOTINE

A. Objective: Determine the effects of adding propylene glycol to the feed filler on the ART process.

B. Results: Propylene glycol is currently being added to the feed filler prior to extraction at the 2% to 4% level. PG is somewhat soluble in SC-CO<sub>2</sub> and has been shown to pass into the CO<sub>2</sub> working vessel during let-down. A potentially dynamic situation could exist if the PG is soluble in liquid CO<sub>2</sub> and is delivered back to the process system during pressure-up. An apparatus was designed and fabricated for studying the solubility behavior of PG in liquid CO<sub>2</sub> at 900 psig and at 24°C. Initial evaluation of the data indicate PG partitions between liquid CO<sub>2</sub> and water at about .011 to .013, strongly in favor of the water. This partitioning is limited, however, by the 0.5 % solubility limit of PG in liquid CO<sub>2</sub>.

C. Plans: Further evaluate data and issue memo.

#### III. LOW NICOTINE

A. Objective: Measure the conductance of SC-CO<sub>2</sub> at ART process conditions.

- B. Results: In cooperation with Development Engineering, found the fluid phase in the ART process to be non-conductive throughout the entire process cycle. This test was to determine if corrosion within the system could be minimized by anodic or cathodic protection.
- C. Plans: Completed and issued report.

#### IV. LOW NICOTINE

- A. Objective: Develop alternate on-line nicotine monitors for the ART process.
- B. Results: Problems with the O-ring seals in the new shorter path length cell have developed. Several O-rings of different hardness and material composition were tried without success. Modifications to the O-ring support structure are underway and are designed so that the physical integrity of the cell is maintained.
- C. Plans: This work is ongoing.

#### V. LOW NICOTINE

- A. Objective: Develop second-generation processing for ART.
- B. Results: Two runs were made using the liquid absorber in order to provide real process water for evaluating the Sepracor Unit held by Chemical Research.
- C. Plans: Work is ongoing.

PROJECT NUMBER: 1708  
PROJECT TITLE: Physical Chemistry and Process Monitoring  
PROJECT LEADER: J. L. Banyasz  
PERIOD COVERED: November 1988

I. OPERATIONS SUPPORT (P. Henderson, in collaboration with the Applied Technology Group)

- A. Objective: Characterization of inks.
- B. Results: Dr. Julius Silver of the Rochester Institute of Technology presented a three-day seminar on printing technology at 20th Street. Dr. Silver has been retained as a consultant.

The capabilities of the ink laboratory have now been considerably extended with the arrival of the printability tester, the rub dryness tester, and the Hunter Labscan colorimeter, in addition to the inkometer and Brookfield viscometer. The ink characterization program has been broadened to include B&H ocher, B&H green, Marlboro black, and L&M maroon in combination with six current production papers. The tests now include setting time, drying time, color coordinates, tack, and viscosity.

- C. Plans: The construction of a simulator to simulate the printing process on the maker is planned.

II. OPERATIONS SUPPORT (J. Crump and A. Closter, in collaboration with the Applied Technology Group)

- A. Objective: Determine the effect of particle size distribution on dynamic viscosity changes that occur in PVA tipping adhesives.
- B. Results: The roller speed study was continued. with the testing of four more adhesives. Of all the materials tested, the best performance was obtained from Ajax adhesive 563-6, currently in use at Louisville. This formulation remained within specifications with regard to viscosity at all times during forty minute idles at various roller speeds. The Fuller "Full Run" adhesive differed from all other materials tested in that it exhibited thixotropic behavior.
- C. Plans: Plans have been made to modify the simulator so as to allow it to simulate the Mark 10A maker.

III. MENTHOL STUDIES (D. Driscoll and T. Van Auken)

- A. Objective: Determine the diffusion coefficient of menthol in cellulose acetate (CA).

- B. Results: The permeability of menthol through CA (unplasticized) film has been determined at 70°C. Only steady state permeability was observed. The experiment will be repeated to ensure no leaks were present. The approach will also be modified so as to allow starting the measurements after a shorter initial time interval in hopes of observing the transient region, which would allow calculation of the diffusion coefficient.
- C Plans: This work is ongoing.



PROJECT NUMBER: 1720  
PROJECT TITLE: Analytical Microscopy  
PROJECT LEADER: V. L. Baliga  
PERIOD COVERED: November, 1988

I. PROJECT ART (Baliga, Henry)

- A. Objective: Characterize changes to the epidermal surfaces of super-critically extracted (SCE) tobaccos and determine degree of expansion or shrinkage.
- B. Results: Three samples were examined for changes in their surface structure, cross sectional thickness, and lateral area expansion or shrinkage. They included a SCE sample, a SCE-liquid CO<sub>2</sub> washed sample, and a SCE-liquid CO<sub>2</sub> washed-DIET expanded sample. Surface erosion was found in the SCE-liquid CO<sub>2</sub> washed sample and the SCE-liquid CO<sub>2</sub>-DIET expanded sample. Sample expansion occurred in the cross section plane with the cross sectional thickness of the SCE-liquid CO<sub>2</sub> washed-DIET expanded sample being over three times thicker than the other two samples. None of the three samples exhibited lateral surface area expansion or shrinkage as measured by counting stomata per unit area<sup>1</sup>.
- C. References:
- Baliga, V., and Henry, B., "Structural Changes in SCE, Liquid CO<sub>2</sub> Washed, and DIET expanded tobacco," memo to Sue Wrenn, October 27, 1988.

II. LOW SIDESTREAM (Henry)

- A. Objective: Examine the structure of cigarette papers and paper additives.
- B. Results: Three samples of production sheet cigarette papers were examined for particle size and distribution of calcium carbonate within the paper. Two papers, both of which contained multiflex MM calcium carbonate, contained small and evenly distributed clumps of calcium carbonate compared to previous examined handsheets<sup>1</sup> made with the same calcium carbonate. The individual particle size was similar to that noted previously at about 0.1  $\mu\text{m}$  to 0.2  $\mu\text{m}$  in diameter compared to the 0.07  $\mu\text{m}$  diameter starting material. The standard barrel-shaped calcium carbonate used in the third production sample was evenly distributed throughout the paper and had an average particle size of 0.2  $\mu\text{m}$  by 2.0  $\mu\text{m}$ . Neither the multiflex MM calcium carbonate nor the standard calcium carbonate coated the flax fibers<sup>2</sup>.
- C. References:
1. Baliga, V., Henry, B., "Structural Characterization of CaCO<sub>3</sub> Handsheets," memo to S. Baldwin, October 13, 1988.

2. Henry, B., "Distribution of Calcium Carbonate in Production Sheets," memo to S. Baldwin, November 9, 1988.

### III. SUPPORT TO OPERATIONS AND R&D (Sanders, Baliga)

A. Objective: Provide analytical support to Operations and R&D.

B. Results:

A customer complaint sample was examined to determine the cause of an off-odor/off-taste quality in Merit Regulars. No foreign materials were found<sup>1</sup>.

Eight white ceramic plates were submitted to be carbon coated. Four of the plates were coated on side one which turned black while the other four plates were coated on side two and they turned light gray. This suggested a difference in the textural quality between sides one and two<sup>2</sup>.

A metal coating of a machine part was examined and found to contain chromium with some silicon debris<sup>3</sup>.

Particles of black material from a flavor mixing tank were examined and found to contain mostly carbon with small amounts of Si, S, Cl, K, Ca, and Fe<sup>4</sup>.

C. References:

1. Sanders, K., "Analytical Microscopy Result Form," to A. Charles, October 24, 1988.
2. Sanders, K., "Analytical Microscopy Result Form," to M. Sprinkel, November 3, 1988.
3. Sanders, K., "Analytical Microscopy Result Form," to P. Grantham, November 14, 1988.
4. Baliga, V., Sanders, K., "Analytical Microscopy Result Form," to S. Ruziak, November 18, 1988.

### IV. SAFETY (Sanders)

A. Objective: Provide instruction to ERT members on various aspects of first aid.

B. Results: One ERT class was given on skull and spinal injuries and the use of backboards.

### V. MISCELLANEOUS (Sanders)

A. Results: An article written by K. Sanders titled "Children of Alcoholics", was published in Virginia Lifeline, November, 1988.

PROJECT NUMBER: 1730  
PROJECT TITLE: Plant, Cell & Tissue Culture Research  
PROJECT LEADER: I. L. Uydess  
WRITTEN BY: M. Shulleeta  
PERIOD COVERED: November, 1988

## I. TOBACCO-IDENTICAL PRESERVATIVES

A. Objective: To develop procedures and to establish microbiological screens for the evaluation of new, nature-identical preservatives as replacements for and/or as adjuncts to propylparaben.

B. Status:

### Phase I Screens:

In previous shake flask experiments it was observed that a **decanol**:paraben mixture (25 µg/ml each component) effectively inhibited bacterial growth while a similar **decanoic acid**:paraben mixture did not. In an effort to assess the relative efficacy of decanol and decanoic acid alone at low (25 µg/ml or less) levels, a series of dose-response experiments employing 12.5, 25 and 50 µg/ml test doses were performed. The results indicated that 25 µg/ml **decanol** was completely inhibitory to the growth of PM #13 (*B. coagulans*) while 50 µg/ml decanoic acid was required to achieve a similar result.

### Phase II Screens:

The comparative efficacies of nonanoic acid (C-9) and dodecanoic acid (C-12) were examined in Park 500 SEL at test doses of 200, 400 and 600 µg/ml. The results indicated that nonanoic acid was as effective as decanoic acid with respect to inhibiting the changes in pH and total CFU that are normally observed in lab-spoiled SEL (1). Dodecanoic acid, however, was not as effective in inhibiting these changes in pH and CFUs as were the C-9 and C-10 compounds.

Mixtures of a fixed concentration of propylparaben and increasing concentrations (doses) of decanoic acid (100 µg/ml paraben to 200, 300 and 400 µg/ml decanoic acid) were also tested in Park 500 SEL. The results indicated that the 100:300 and 100:400 mixtures effectively inhibited changes in pH and/or the increase in CFUs associated with spoilage. However, the efficacy observed appeared to be proportionate to the level and activity of decanoic acid only. For example, the 100:300 mixture was as efficacious as 300 µg/ml decanoic acid alone (with respect to measured parameters).

### Phase III Screens:

In an effort to determine the optimum temperature for spoilage (and thus for Phase III fermentor screens as well) additional experiments were conducted at 42° and 45°C in Park 500 SEL in 5-liter fermentors (experiments at 37° and 47°C were conducted previously). Initial results indicated that 42°C appears to be the optimum temperature at

which this type of spoilage occurs (increase in pH and organic acids, decrease in nitrate-nitrogen, etc.). At this temperature the total CFUs were notably higher as well. Of additional interest was the observation that the effectiveness of decanoic acid was even more dramatic at 42°C than at 37° or 45°C due to the higher growth rate associated with this temperature. The efficacy of bactericidal compounds like decanoic acid has been shown to be related to the relative physiologic state (growth rate) of the target organism: the higher the growth rate, the more pronounced the antimicrobial effect (2).

**Miscellaneous:**

Hand made cigarettes from the pilot plant sheet runs of October 10, 1988 employing decanoic acid have been submitted for subjective evaluation.

**C. Conclusions:**

1. In Phase I testing decanol has been demonstrated to be a more potent antimicrobial agent than decanoic acid.
2. In Phase II screens nonanoic acid (C-9) has proved as effective as decanoic acid (C-10) at the doses tested.
3. Combinations of paraben and decanoic acid effectively prevented changes in pH and increases in CFUs associated with lab-spoiled SEL.
4. The optimum temperature at which to conduct Phase III fermentor screens has been determined to be 42°C.

**D. Plans: December, 1988**

1. Phase II (shake flask) and Phase III (fermentor) studies will be conducted in conjunction with M. Hofer of FTR.
2. Further evaluations in Phase II and Phase III test systems of the C-9 through C-12 fatty acids and/or alcohols alone and in concert with parabens will be conducted.

**E. References:**

1. Lyle, J. and Weissbecker, L. (memo in preparation).
2. Uydess, I.L., Shulleeta, M. and Tenhet, S., "Decanoic Acid: Static or Cidal Inhibitor of Bacterial Growth?", PM Memo to Dr. R.W. McCuen, October 25, 1988.

PROJECT NUMBER: 1752  
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke  
PROJECT LEADER: G. Vilcins  
PERIOD COVERED: November, 1988

## I. TANDEM MASS SPECTROMETRY

- A. Objective: To establish a mass spectrometry center with the state of the art capability in tandem, high resolution, and soft ionization mass spectrometry.
- B. Results: In order to develop methodology for the characterization of complex lipids by utilizing soft ionization and tandem mass spectrometry, a trip was made to the Midwest Center for Mass Spectrometry. Lipid extracts from tobacco before and after ART processing, samples of residue from the ART process, and known lipid mixtures were prepared for FAB and NCI analysis. Some samples were analyzed directly. Others were fractionated into acid, basic and neutral fractions and in some cases simple derivitization was employed to enhance ionization. The samples were analyzed using the MS-50 triple analyzer mass spectrometer.
- C. Conclusions: Evaluation of data is in progress. Particularly significant results include information on fatty acid composition of the ART residues and differences in complex lipids in tobacco before and after the ART process. Work with known fatty alcohols demonstrated that nicotinyl chloride hydrochloride derivatives are useful for enhancing specific structural information for long chained alcohols.
- D. Plans: To evaluate the data of the ART residue samples and the known long chained alcohols. To prepare reports on data obtained and to explore some of the results with further experimentation.

## II. SMOKE ANALYSIS

- A. Objective: To compare the smoke chemistry of control and ART extracted filler.
- B. Results: Mainstream smoke from weight selected control (x6d8dej) and Art (x6d8dek) cigarettes were fractionated and compared to reveal any difference relating to the extraction process. Since the objective is to compare fillers, cigarette construction effects were eliminated by removing the CA filter and taping the ventilation holes prior to smoking. Mean area counts (from four replicates) from a set of target compounds in each fraction were compared. In the basic fraction, smoke from ART filler contained more volatile bases than that from control such as methylpyrazine; 2,6-dimethylpyrazine; 2,3,6-trimethylpyrazine; 3-methylpyrrolpyrazine; dimethylaminoacetonitrile, dimethylpyridines, ethylmethylpyridines, quinoline, and a minor alkaloid, nornicotine. The presence of these excessive bases was probably due to the high level of ammonia in the ART filler which

induced Maillard reaction with amino acids. The smoke from control showed higher amount of 3-vinylpyridine, 3-ethylpyridine, myosmine, nicotyrine, 2,2-dipyridine, cotinine, and some acyl alkaloids which reflected the difference in nicotine concentration between ART and control fillers. In the acidic fraction, volatile fatty acids such as 2-methylbutyric acid, valeric acid, beta-methylvaleric acids were reduced in the ART smoke. Some simple phenols such as cresols, guaiacol, 2-vinylphenol, and 4-vinylguaiacol were higher in the smoke of ART filler. Higher fatty acids showed comparable levels in both samples. In the neutral fraction, ART cigarette was consistently lower in the compounds identified such as furfural, limonene, 1-indanone, solanone, megastigmatrienones (4 isomers) and neophytadiene.

- C. **Conclusions:** The mainstream smoke of ART filler contained more volatile bases, some phenols, less volatile fatty acids, and less neutral compounds than that of control filler.

D. **References:**

Hsu, F, Coleman, S., and A. Oti, "Smoke Chemistry - ART," memo to B. Handy, October 31, 1988.

### III. THERMOGRAVIMETRIC ANALYSIS (TGA) OF CARBON DIOXIDE IN DOLOMITE

- A. **Objective:** The purpose of this experiment is to develop methodology for the determination of percent composition of carbon dioxide in the mineral Dolomite.
- B. **Results:** Six Dolomite samples were analyzed using a Perkin-Elmer TGA-7 analyzer. The samples were scanned from 25 C to 1025 C at a scan rate of 70 C/minute. The samples yielded from 31.0 percent to 31.5 percent weight loss from 421 C to 955 C which was assumed to be carbon dioxide since most carbonates decompose at this temperature range. The average weight of the carbon dioxide for the six samples was 31.3 percent.
- C. **Conclusions:** Based on the precision of the results, the relative short time necessary for analysis, and the ease of interpretation, TGA should be considered for the analysis of carbon dioxide in Dolomite and similar samples (i.e. samples containing carbonates and hydrates).
- D. **Plans:** The TGA method of analysis of carbon dioxide in Dolomite will be compared with previous methods. Future plans include structural elucidation and determination of percent composition of various carbonates and hydrates in Dolomite with data obtained from elemental analysis from Atomic Absorption and decomposition studies of Dolomite and pure carbonates and hydrates from TGA.

IV. THERMAL CHROMATOGRAPHY-MASS SPECTROMETRY

- A. Objective: To determine the effect of pet ether extraction on ART tobacco.
- B. Results: Comparisons are being made on feed filler, ART filler, and pet ether extracted ART filler using the TC/MS instrument.
- C. Conclusions: Data are under evaluation.

PROJECT NUMBER: 1754  
PROJECT TITLE: Spectroscopic Studies of Tobacco and Smoke Components  
PROJECT LEADER: J. B. Wooten  
WRITTEN BY: R. L. Bassfield  
PERIOD COVERED: November, 1988

SOLUTION NMR

- A. Objective: Use NMR to determine the optical purity of nicotine.
- B. Results: Experiments using  $\beta$ -cyclodextrin as a chiral shift reagent to produce chemical shift differences in racemic nicotine were not successful.
- C. Plans: Additional experiments are planned where the nicotine will be protonated in order to facilitate the inclusion by the cyclodextrin. Another set of experiments using a derivative of tartaric acid as the chiral shift reagent will also be explored.
- A. Objective: Use NMR to confirm the structure and purity of alkyl pyrazines used in flavor studies.
- B. Results:  $^1\text{H}$  and  $^{13}\text{C}$  data were collected on an additional series of alkyl pyrazine derivatives. This series of compounds proved to have the correct structure and was of a high degree of purity (95+%). The trace amounts of water found in the previous series appears to be removed from this series.



PROJECT NUMBER: 1757  
PROJECT TITLE: Analytical Flavor Specifications  
PROJECT LEADER: M. L. Zimmermann  
PERIOD COVERED: November, 1988

#### ANALYTICAL FLAVOR SPECIFICATIONS

A. Objective: To develop analytical and sensory specifications for incoming flavors and materials for use at the Flavor Center and other QA facilities.

B. Results:

The last sample for the second vendor has been received, however, evaluation of these materials has been suspended to handle the analysis of the PMI shipment lot samples. A coordinated effort by members of the Analytical Research Division has greatly aided this effort. A sample was sent out for mercury analysis as we do not have this capability. All reference materials for this work have been located.

An ART humectant sample was submitted for purity specification.

C. Plans: Complete specifications for the second vendor and continue with the work on the PMI samples.

PROJECT NUMBER: 1758  
PROJECT TITLE: Tobacco Cell Wall Research  
PROJECT LEADER: G. H. Bokelman  
PERIOD COVERED: November, 1988

I. SIDESTREAM REDUCTION (B. Rogers, S. Tafur, G. Bokelman and S. Baldwin)

- A. Objectives: (1) Prepare cigarette paper, acceptable both in appearance and porosity, on the pilot-scale paper machine at the University of Maine and (2) prepare and evaluate (for reduction in sidestream smoke) paper handsheets that contain coatings and/or inorganic fillers with different physical and chemical properties.
- B. Results: Additional experiments were conducted using the paper machine in the Chemical Engineering Department at the University of Maine, which led to significant improvements in tightness, appearance and run-ability of the paper (1). Improvements in preparing a less permeable paper were attributable to use of a white water recirculation system (allowing us to recover fines) and utilization of a Claflin refiner, after the valley beater, which further reduced fiber weight length. The formation, and thus appearance, of the paper was greatly improved when an Impco screen (to remove clumps of long fibers) was inserted between the machine chest and head box. The run-ability of the paper was improved by the use of dual squirt trimmers on both sides of the paper web. By widening the gaps between the paper and the excess material on either side of it the frequency of breaks at the couch was markedly decreased. In our most successful run to date, a partial bobbin of cigarette paper was prepared with a porosity of 7 Coresta, a basis weight of 38.8 g/m<sup>2</sup> and a (Multifex MM) calcium carbonate content of 27.5%.

A laboratory scale coater was obtained and procedures were developed for sizing and coating. Handsheets prepared at the University of Maine in September and October were treated with potassium succinate, and cigarettes were made from them. Sidestream light extinction data are pending on these cigarettes.

Samples of low porosity, chemical free paper obtained from Kimberly Clark were treated with 5% potassium succinate and coated with four different samples of carboxy methylcellulose (CMC) containing different cations. Calcium, magnesium, potassium, and ammonium ions are present in these samples in full or partial substitution for the sodium ion. The CMC's were applied at a 0.5% level. Cigarettes made from these papers will be evaluated subjectively against pure sodium CMC.

A total of 21 samples of handmade cigarettes have been prepared from cigarette paper handsheets and submitted for single port sidestream light extinction measurements. These were primarily cigarettes made with papers with varying particle sizes of CaCO<sub>3</sub> at varying porosities. Cigarettes with papers made with Mg(OH)<sub>2</sub>, dolomite and MgCO<sub>3</sub> were also submitted. Preliminary results continue to show that MgCO<sub>3</sub> looks promising for reduction of sidestream visibility.

Work has begun to examine the use of "tobacco solubles" for producing cigarette paper which reduces sidestream visibility and possibly improves mainstream and sidestream subjectives. The initial research has involved producing aqueous extracts of ART tobacco at room temperature and by Soxhlet extraction.

- C. Plans: In addition to the continuing preparation and evaluation of handsheets (with and without coatings) and handmade cigarettes, an evaluation will be made of the use of two head boxes to produce bilayer sheets on the paper machine at the University of Maine.

D. Reference:

1. Bokelman, G. H., memo to R. N. Ferguson, "Results from the November Trip to the University of Maine," November 15, 1988.

II. BLEND COMPOSITIONS OF PRIME AND SALEM LIGHTS (HONG KONG)

(G. Bokelman, J. Stimler and General Analytical)

- A. Objective: Determine the blend compositions of Prime, a new cigarette produced by JTI for the Japanese market, and Salem Lights (Hong Kong).
- B. Results: The blend composition of Prime was found to be very similar to those of SomeTime Lights (from JTI) and Marlboro Lights (Japan) (1).

Based on comparison with an earlier study, it appears that there are three different formulations of Salem Lights for the U.S., Japan and Hong Kong (2). The two brands marketed in Asia have increased contents of bright lamina and decreased contents of burley lamina and Oriental leaf compared to their U.S. counterpart. Not surprisingly, the Japanese version of Salem Lights has the highest stem content.

- C. Plans: Preparation of an updated tobacco database, for use with future blend composition requests, is now in progress.

D. References:

1. Bokelman, G. H. and J. O. Stimler, memo to S. B. Nelson, III, "Blend Composition Analysis of Prime," November 17, 1988.
2. Bokelman, G. H. and J. O. Stimler, memo to Linda Vinson, "Blend Composition Analysis of Salem Lights (Hong Kong)," November 17, 1988.

PROJECT NUMBER: 1902  
PROJECT TITLE: Tobacco Microbiology  
PROJECT LEADER: D. J. Ayers  
WRITTEN BY: J. B. Jones  
PERIOD COVERED: November, 1988

#### I. ART FILLER 12 WEEK STUDY

- A. Objective: To determine the effects of storage on the microbial concentration of ART filler material at 35°C/80% RH and 25°C/60% RH.
- B. Results: Bacterial counts showed no increases, over initial counts, for the data accumulated from experiments 1-5 after 12, 8, 8, 8, 4 weeks of storage, respectively. The mold counts did not exceed acceptable laboratory limits but the yeast counts exceeded acceptable laboratory limits (> 80 mold/yeast colonies per gram) (1) in all 5 experiments to date (2).
- C. Plans: Complete current study.
- D. References:
1. Crockett, E. A. The Microbial Quality Improvement Program (MQIP) As Conducted in the OC Semi-Works Primary Facility. Special Report #87-155; 1987 December 21.
  2. Jones, J. Notebook No. 8590, pp. 73, 75, 77-80, 83-84, 87.

#### II. MONOPOTASSIUM CITRATE STORAGE STUDY

- A. Objective: To determine the effects of storage on the microbial load of monopotassium citrate at 25°C and 45°C.
- B. Results: No bacterial, mold, or yeast colonies have been detected to date in the sample at 25°C or 45°C. Samples were tested on a daily basis for the first week and on a weekly basis for the second week to date.
- C. Plans: Continue the testing.
- D. Reference:
1. Gaines, O. Notebook No. 8690, pp. 78-79.

#### III. BACTOMETER CALIBRATION CURVES

- A. Objective: To generate calibration curves for determining the number of bacteria present in a sample using the Bactometer.
- B. Results: Data will be complete by the end of November for the Oriental tobacco calibration curve.

C. Plans: Complete data collection for the completion of calibration curves for bright tobacco and a typical, full-flavored blend.

D. Reference:

1. Chadick, D. Notebook No. 8625, pp. 158-161.

#### IV. CTB AND BURLEY SPRAYED BURLEY STORAGE STUDY

A. Objective: To determine if the addition of class tobacco dust (CT) to the Regular Burley Spray (RBS) would enhance microbial development on the Burley Strip after 12 weeks of storage at 35°C with 80% RH.

B. Results: CTB and RBS sprayed Burley tobacco from Louisville and Stockton Street have been microbially examined after 8 weeks of storage. Bacterial, mold, and yeast counts are within laboratory limits to date.

C. Plans: Complete week 12 of storage.

D. Reference:

1. Chadick, D. Notebook No. 8625, pp. 133-138, 141-142, 149, 154.

PROJECT NUMBER: 1904  
PROJECT TITLE: Tobacco Physiology and Biochemistry  
PROJECT LEADER: H. Y. Nakatani  
PERIOD COVERED: November, 1988

## I. LOW NICOTINE STUDY

- A. Objective: To investigate the biochemistry of the nicotine biosynthetic pathway at putrescine N-methyltransferase (PMT) and specifically to isolate PMT from tobacco root extracts.
- B. Status: The roots from the group 15 tobacco plants were harvested and stored at  $-80^{\circ}\text{C}$ . The roots from group 14 and the remaining roots from group 13 have been processed to the ammonium sulfate (40-65%) stage. In addition two extracts (ammonium sulfate) have been prepared from the group 15 harvest (1). Bulk ammonium sulfate samples have been processed through the phenyl-Sepharose column (2).

Additional studies have been conducted with the S-adenosyl-homocysteine (SAH) affinity matrix for the purification of PMT. It appears that PMT may bind to this matrix by two modes of interactions a) affinity and b) ion-exchange or other mechanism(s) (2). PMT and other methyltransferases which are bound in the affinity mode are eluted from this column with 1 mM SAM; PMT and other proteins bound in the latter mode(s) are then removed by elution with 0.5 M NaCl (2). An SAH-column which was blocked with ethanolamine rather than mercaptoethanol was not found to function better than the mercaptoethanol blocked matrix (5,6). Additional studies were also conducted with the  $\omega$ -aminoethyl agarose (AEA) matrix. This latter column was found to be promising for the purification of PMT (5,6). A SAM-eluted PMT fraction from the SAH column was applied to the AEA column. The peak fraction having PMT-activity upon elution with 5 mM putrescine showed a specific activity of 51,000 units (nmoles/mg protein at  $30^{\circ}\text{C}$  for 30 min.) (5,6). Two preparations of PM extracts (PM 84 and 85) from the ammonium sulfate stage were processed through the following series: phenyl-Sepharose, SAH-affinity and AEA-affinity columns. The pooled samples after the AEA-affinity column in both cases showed an increase in specific activity of about 500-fold compared with the ammonium sulfate stage. Moreover, the specific activities of the pooled PMT-active fractions were  $>11,000$  (1,2,3,5,6). A set of samples from the various purification steps from PM 84 were prepared and sent to PDI (Protein Database Incorporated) for 2-D examination (3). SDS-Page examination from these samples were also obtained (3). A sample from the SAH stage was submitted for  $^3\text{H}$ -SAM photoaffinity labeling (Dr. Bruce Davies).

Further studies were continued with the SAH column to obtain greater amounts of PMT for processing through the AEA column. Following elution of PMT-activity with SAM, the column was washed with dialysis buffer. PMT-activity was again eluted with SAM after applying additional PMT-active samples (phenyl-Sepharose stage) to this column (5,6). This elution scheme decreases the processing

time to obtain PMT from the SAH column without having to recycle the SAH matrix completely.

A number of miscellaneous studies have also been conducted. Examination of the N-methylputrescine affinity (NMP) matrix was continued. However, no increase in specific activity of PMT over the starting material was obtained for any of the NMP-affinity matrices tested. These results precluded its use at this time although recovery of PMT activity ranged from 6-25% of the applied sample (from prep 1) (4). A longer separating gel was examined in the ELFE system in an attempt to increase the resolution of the native gel system. This was not helpful since the increased electrophoresis time resulted in the loss of PMT activity (3). The Rotofor is being examined as a possible means of resolving the most purified samples by isoelectric focusing. 1 mM SAM was not found to increase the stability of PMT preparations over a three day time course at 4°C (3). Dr. Walter P. Hempfling has also reconfirmed that the molecular weight of PMT is ~60,000 daltons by using a P100 BioGel matrix (Bio-Rad) gel filtration column. This is consistent with previous results obtained with the Pharmacia Sephacryl gel filtration columns (4). More sensitive methods for assaying proteins are being investigated: a fluorescence method using fluorescamine and a Protein-Gold reagent (Integrated Separations System) (4).

- C. Plans: Continue to process roots to the 40-65% ammonium sulfate stage. Prepare enriched PMT samples from the ammonium sulfate stage using phenyl-Sepharose and SAH and AEA-columns. Determine efficacy of one of the more sensitive protein assay methods for use in our studies. Evaluate other classical chromatographic methods to yield a homogeneous PMT preparation.

D. References:

1. Dunn, R. L. Notebook No. 7899.
2. Malik, V. Notebook No. 8542.
3. Davies, S. Notebook No. 8694.
4. Yu, T. Notebook No. 8381.
5. Mooz, E. D. Notebook No. 8599.
6. Crockett, E. Notebook No. 8563.

PROJECT NUMBER: 2106  
PROJECT TITLE: Cigarette Performance and Design  
PROJECT LEADER: R. W. Dwyer  
PERIOD COVERED: November 1988

I. FILTER RESEARCH AND DEVELOPMENT (J. Kao)

- A. Objective: Evaluate the influence of tobacco-rod butt length on filter efficiencies..
- B. Results: A mathematical model has been developed which accounts for the effect of tobacco-rod butt length on filter efficiency. Eastman has provided us with the raw data required to develop such a model. Their data include TPM deliveries as functions of puff volume and puff position for cigarettes with a variety of filters. These data, as well as in-house results, have been used to model the effects of filter pressure drop, dimensions, tow item, ventilation level, and puff position on efficiencies.
- C. Conclusions: As the butt length decreases, the smoke removal efficiency of the filter increases, most likely due to increased condensation of vapors on the filter. The increase in efficiency is a function of the reciprocal of the rod butt length.
- D. Plans: This model will be incorporated into the expert-system based cigarette design model.

II. CIGARETTE DESIGN COMPUTER MODEL (D. Leister and L. Watt)

- A. Objective: Provide the Filter R&D Project with an expert-system based cigarette-design system.
- B. Results: The hardware and software to run the cigarette-design-and-performance system have been installed in the Filter R&D lab at the O.C. Instructions for using this prototype system were provided as well as a demonstration run.
- C. Plans: This system will be refined on a continuing basis by incorporating our on-going research results, and also by enhancing its ease of use. At this time we are adding the capability of selecting tow items for concentric filters and predicting their effects on cigarette deliveries.

III. FILTER VENTILATION (D. Simpson)

- A. Objective: Predict filter ventilation levels based on the permeabilities of tipping papers and plug wraps.
- B. Results: We have had cigarettes fabricated with a variety of tipping papers and plug wraps, all other design parameters being held constant. These models were made with zero, one, or two anchor lines on the plug wrap. Additionally, experiments have



been performed in which the pressure drops as functions of flow rate were measured for radial flow through CA filters.

- C. **Conclusions:** The presence of anchor lines has little effect on the level of filter ventilation. The contribution of radial flow through the filter plug directly under the vents is relatively small, but needs to be accounted for in predicting filter ventilations.
- D. **Plans:** These results will be incorporated into a mathematical model for predicting ventilation levels. Further experiments are in progress for evaluating the influence of different tipping machines on filter ventilation levels.

#### IV. TPM GENERATION EFFICIENCIES (L. Goodwin and B. Dwyer)

- A. **Objective:** Evaluate the effects of blend composition on the generation of TPM.
- B. **Results:** We have developed equations which allow the calculation of the weight of tobacco consumed during a single puff. We have also developed equations for estimating the weight of TPM generated at the coal during a single puff. Using these relationships, the cigarette data in the CI data base have been evaluated for TPM generation efficiencies, i.e., the ratio of generated-TPM-weight to consumed-tobacco-weight.
- C. **Conclusions:** The TPM generation efficiencies of cigarettes vary from brand to brand over a relatively small range. Currently we are examining the data base to see if correlations exist between the generation efficiencies and the blend compositions of the CI brands.

PROJECT NUMBER: 2500  
PROJECT TITLE: Fundamental Chemistry  
PROJECT LEADER: J. I. Seeman  
PERIOD COVERED: November 1988

I. FLAVOR/ODOR CHEMISTRY (Houminer, Paine)

- A. Objective: To develop new technologies for smoke deliveries of desired flavorants; to prepare new substances for flavor/odor evaluation.
- B. Results and Plans: Work continues on the preparation of polyMIC analogues. The bis(menthylcarbonate) of 2,5-dimethylhexan-2,5-diol was successfully prepared; other related reactions did not yield the desired products. A second kinetic run of the thermolysis of six pyrazineethanols (pyrazine release agents) has been completed and analysis of the results is in progress. The synthesis of several dialkylpyrazines is being undertaken, for flavor studies and for laser jet spectroscopic studies.

II. CHEMICAL PHYSICS STUDIES OF TOBACCO CONSTITUENTS (Seeman)

- A. Objective: To obtain structural information about important tobacco constituents/flavorants; to develop information on cluster formation and chemical reactions in clusters.
- B. Results and Plans: Plans are being formulated for future studies of chemical reactions, including the design of a dual beam apparatus. Clusters of analogues of nornicotine and nicotine with water, methane, and carbon dioxide are being studied.

III. REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS (Howe, Secor, Seeman)

- A. Objective: To develop techniques to remove nicotine and other tobacco alkaloids from aqueous tobacco processing fluids to the exclusion of all other components.
- B. Results and Plans: Two Sepracor personnel installed the bench scale membrane apparatus. Two nicotine separations were performed using second generation-ART water absorber liquid prepared specifically for this study by J. Dobbs. GC analysis by C. Tatum indicated >90% removal of the nicotine from the absorber liquid and transference to the back extract. A series of tartaric acid esters were prepared as novel, proprietary liquid ion exchangers for the membrane process. Preliminary partition coefficient data indicates that at least two of these esters may be excellent candidates.

IV. MISCELLANEOUS (Secor)

- A. Results: Together with R. Izac, >500 mg of 4'-(2-hydroxyethyl)-nicotine was prepared on special request for the Biochemical Research Division.

PROJECT NUMBER: 2501  
PROJECT TITLE: Smoke Chemistry  
PROJECT LEADER: R. Comes  
WRITTEN BY: D. R. Douglas  
PERIOD COVERED: November 1988

## I. SIDESTREAM SMOKE

**A. Objective:** Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream gas phase and semivolatiles; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of proprietary products.

**B. Status:** 1.) Methods development for analysis of sidestream smoke components continues. 2.) Construction and testing of instrumentation to monitor visibility of sidestream smoke has been ongoing. 3.) Studies have proceeded using the CORESTA sidestream measurement apparatus in its current state of refinement. Selected samples were run to support this effort.

**C. Results:** 1.) A second-generation Tekmar desorber has been received. The original model has been reconnected to the valve-operated heart-cutting gc's. Extensive plumbing and pressure-balance work has resulted in a preliminary ability to transfer portions of sidestream gas phase to the second gc column for further separation and identification by mass spectrometry. A second instrument and method has been prepared and developed for analysis of silica gel fractions of semivolatiles of sidestream smoke. This frees the previously used instrument for other work described below. 2.) The 17-port smoking machine has been removed from the conditioned lab. It has been replaced with the 8-port instrument, which has been redesigned, rebuilt, and is undergoing testing. 3.) Control labelled-tobacco cigarettes and balsa-core cigarettes were tested with the CORESTA apparatus. Results from this work are inconclusive and will require additional experiments, possibly using other types of analyses to compare with results obtained to date.

**D. Plans:** 1.) The currently used Tekmar unit will be exchanged for the new one with the help of the Tekmar service representative. This will permit us to utilize his expertise in installation, and give him a first-hand look at our use of the instrument. The older instrument will then be returned to the factory for retro-fitting with current generation hardware. Additional development work on valve-switched heart-cutting capillary gc will determine whether this is a viable technique for this analysis. If not, pressure-switched heart-cutting will be investigated. The auto-sampler gc will be used to examine semivolatiles from control and experimental, low-sidestream cigarette models. 2.) Improvement of the 8-port smoking apparatus will be continued with the objective of providing a reliable instrument for measurement of visibility of sidestream smoke so that the instrument and method can be duplicated in other labs. 3.) Additional

experimental work and study of data already gathered will be done to clarify the effect of cigarettes with novel constructions on sidestream delivery.

## II. SIDESTREAM SMOKE CHAMBER

A. Objective: Design and construct an environmentally controlled chamber to measure selected components of sidestream smoke.

B. Status: Final authorization of the chamber project has been given. Final specifications for some instruments have been reviewed and they have been ordered. The electronic/computer control and data acquisition capabilities for the chamber are being attended to with the help of the divisions responsible for this portion of the work. Final plans are being prepared by the vendor for construction of the chamber.

C. Results: Cooperative work among responsible parties in R&D and with vendors has moved this project from the planning stage to beginning the assembly and operation of the chamber.

D. Plans: Meet with chamber vendor to review final plans; visit vendor's facility to observe chamber construction; approve final chamber design and authorize construction; request lab modifications including hood installation and other work necessary to accommodate the chamber; bench-test instruments as they are received before installation in chamber; provide hardware and software for data acquisition and archival in database, and for chamber and instrument control.

## III. MISCELLANEOUS

Analysis of nicotine/alkaloids is being carried out by capillary gc to support various aspects of the ART project.

Analyses of smoke and extracts of un-burned Premier cigarettes have been done by capillary gc-ms. Identifications of many components in the product and its smoke have been reported by memo. Neutron radiography has also been used to study this product.

The new Finnigan mass spectrometer is operational and familiarization with its use is continuing.

PROJECT NUMBER: 2525  
PROJECT TITLE: Tobacco Chemistry  
PROJECT LEADER: R. R. Izac  
PERIOD COVERED: November, 1988

#### I. NATURAL PRODUCTS CHEMISTRY

- A. Objective: To isolate, identify and/or analyze natural compounds with major emphasis on tobacco and tobacco products.
- B. Results: 1.) A sample of the contents of a dry ice trap collected from Art treated tobacco (samples obtained from S. Wren) is being analyzed. 2.) As part of a collaboration with Project 6906, the separation of Burley S1 (BuS1) fraction using a gel column (TSK Toyopearl HW 40F) was completed. About ten grams of each of the two fractions were supplied to Project 6906. Several fractions obtained by a partitioning scheme (D. Magin) were analyzed for comparison to the samples obtained from the gel column. 3.) Several samples of Dryopteris filix-mas were obtained for AR.
- C. Plans: Continue analysis of causative agent from the Art tobacco cigarette paper and continue separation of BuS1 using a reverse phase column.
- D. References:
1. Izac, R. Notebook No. 8632.
  2. Core, M. Notebook No. 8608.

#### II. LOW NICOTINE

- A. Objective: To examine characteristics of unextracted nicotine.
- B. Results:
- 1.) Grafted tobacco on a tomato rootstock appears to contain only extractable nicotine. None of the tobacco on tomato rootstock grafts have taken and a new series of grafts are being planed.
- C. Plans: Continue to investigate genetic and cultural factors which influence the amount of unextracted nicotine in tobacco materials.
- D. References:
1. Izac, R. Notebook No. 8632.
  2. Bass, R. Notebook No. 8607.
  3. West, G. Notebook No. 8559.

#### III. GREENHOUSE STUDIES

- A. Objective: To maintain the R&D greenhouses, to conduct plant research studies and to provide greenhouse-grown tobacco materials for support of other R&D programs.

- B. **Results:** 1.) The hydroponic Burley 21 plants of Group 15 have been harvested. Group 16 has been started. These plants are being grown for Project 1904. 2.) A second batch of the Burley 21/N. *glutinosa* grafts were harvested. The purpose of this is to provide a plant which boosts the production of nornicotine. 3.) The greenhouse maintenance operations including seeding, transplanting, nutrient solution preparation and other cultural tasks were completed. 4.) The experimental flue-cured and Oriental tobacco grown at Whiteville Research Station has been received and distributed to Project 6908.
- C. **Plans:** Maintain production of fresh root tissue by hydroponic culture.
- D. **References:**
1. Bass, R. Notebook No. 8607.
  2. West, G. Notebook No. 8559.

#### IV. SUPPORT ACTIVITIES

- A. **Objective:** To provide requested assistance for special projects.
- B. **Results:** 1.) An investigation into the synthesis of poly(isopropenyl alcohol) was completed. Poly(isopropenyl acetate) when reacted with fresh sodium methoxide in methanol to gives poly(isopropenyl alcohol) in good yield. Poly(isopropenyl alcohol) when reacted with excess menthyl chloroformate fails to provide the expected poly(menthyl isopropenyl carbonate), polymic, but yields recovered alcohol and dimethylcarbonate. 2.) The analysis for the 1988 R. G. Seed Co. Breeding Lines Test have been completed and the results forwarded. Cigarettes have been prepared from filler obtained from the 1988 Flue-Cured Regional Farm Test. 3.) The 1988 N. C. Flue-Cured Variety Evaluation and the Virginia Evaluation Display were attended with about 840 samples evaluated. 4.) A total of twenty-one handmade cigarettes, some containing a central core of balsa wood, were fabricated in various configurations. 5.) Several aqueous and decane solutions of nicotine were analyzed for nicotine for H. Secor.
- C. **Plans:** Continue to investigate preparation of poly(isopropenyl alcohol) and polymic.
- D. **References:**
1. Izac, R. Notebook No. 8632.
  2. Bass, R. Notebook No. 8607.

PROJECT NUMBER: 6502  
PROJECT TITLE: Environmental Tobacco Smoke  
PROJECT LEADER: C. E. Thomas  
PERIOD COVERED: November, 1988

## I. MAINSTREAM AND SIDESTREAM SMOKE STUDIES

- A. Objective: Develop a method, using the tunable diode laser system (TDL), for the determination of methanol in MS and SS tobacco smoke.
- B. Results: The TDL system used previously to study acrolein deliveries in MS and SS tobacco smoke has been used to quantify methanol in MS smoke. The selectivity and sensitivity of the method were optimized using known amounts of methanol vapor in nitrogen. In initial tests, a monitor #25 was found to deliver approximately 250 ug/cigt. methanol in the MS smoke. A large difference was found between the methanol delivery of the first puff versus the last puff. The first puff delivered 2-4 ug while the last puff delivered about 100 ug. The limit of detection for the method is 0.1 ug/puff.
- C. Conclusions: A method has been developed for MS puff-by-puff methanol in cigarette smoke using TDL spectroscopy. Work on methanol deliveries of submitted samples was completed.
- D. Plans: Additional studies will examine the effects of cigarette blend components on methanol deliveries. The amounts of methanol in the fillers will be measured prior to lighting the cigarettes.
- E. References:
1. Parrish, M., Notebook 8729, pp. 1-2.
  2. Lipscomb, J., Notebook 8703, pp. 12-19.

## II. AMBIENT MONITORING OF ENVIRONMENTAL TOBACCO SMOKE

- A. Objective: Test and evaluate new methods and instrumentation for the measurement of environmental tobacco smoke (ETS) components and incorporate useful procedures into future ETS studies.
- B. Results: A recent visit was made to ACVA Atlantic Inc. in Fairfax, Va. to inspect several different instruments they use to measure indoor air quality. Of interest were an infrared CO<sub>2</sub> monitor and a piezobalance instrument for measuring airborne particulates. The CO<sub>2</sub> monitor would supplement the PASS case which currently is unable to measure carbon dioxide levels. The piezobalance is the method of choice for measuring low levels (5-50 ug/m<sup>3</sup>) of particulates. It is often used in place of collection devices for measuring RSP. More recently, Dr. Stetter, president of Transducer Research (TRI), visited PM R&D and discussed new CO sensors for use in the PASS cases. These new sensors will be less prone to drift in low humidity conditions



such as on commercial aircraft and will have at least twice the usable lifetime. He also discussed  $\text{NO}_2$  and  $\text{SO}_2$  electrochemical sensors which could be easily incorporated into the PASS cases.

- C. Conclusions: The instruments used by ACVA have been used in the field very successfully and are both portable and compact. The carbon dioxide monitor was well suited for our needs and would be useful in future ETS studies. The new CO sensors from TRI should solve the problems of the CO monitors when used in aircraft.
- D. Plans: Evaluate the piezobalance method of particulates versus standard RSP measurements. This would include the recommendation to acquire a piezobalance similar to the type used by both the Environmental protection agency and ACVA Atlantic. Verify that the new sensors from TRI perform according to their specifications. Study the possibility of adding relative humidity,  $\text{NO}_2$ , and  $\text{SO}_2$  sensors to the PASS cases.
- E. References:
1. Koller, K., Notebook 8700, p. 23.
  2. Parrish, M., Notebook 8729, p. 2.

PROJECT NUMBER: 6505  
PROJECT TITLE: Special Investigations/Methods Development  
PROJECT LEADER: D. F. Ingraham  
PERIOD COVERED: November, 1988

I. PROJECT ART

A. Objective: Provide analytical support to project ART.

B. Results:

Some changes have been implemented in an attempt to increase the precision and accuracy of the GC method for the analysis of feed and extracted tobaccos. These have included the use of more standards in the expected range of nicotine concentrations and more extensive use of monitor samples. The analysis of the monitor samples will be used to compute the precision and reproducibility of the method. These data will also be used to isolate the GC precision from the precision of the overall method.

Development of an HPLC method for the determination of nicotine continues. The purpose of this method is to serve as an independent method which can be used as a referee for the GC method. Three different extraction systems have been tried and all three are acceptable for feed tobacco, but result in unacceptable interferences for extracted tobacco. Several solid phase extraction schemes were tried to clean up the sample with no success.

C. Plans: Continue analytical support on an as needed basis. Continue to try different clean-up procedures for the HPLC method. After a sufficient number of samples have been run, subject the monitor data to statistical analysis to determine the precision of the GC method.

II. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIAL

A. Objective: To provide headspace analyses for residual solvents from packaging materials and develop a QA method for the routine analysis of packaging materials.

B. Results: QA continues to analyze routine packaging material for residual solvents while Project 6505 is analyzing samples from new products and other non-routine requests such as inks. Training is being provided to some of the suppliers of packaging materials in the setup and execution of the headspace/GC method.

C. Plans: We will continue to analyze non-routine samples and provide support to QA as needed.

### III. MATERIALS EVALUATION

- A. Objective: To identify components of commercial products prior to their use at PM facilities.
- B. Results: Samples analyzed this month included belting material, static free bags for computer parts, several cleaners, spray-on insulation, sealants, lubricants, and pack overwraps. Four presentations were made to PM staff in Louisville.
- C. Plans: Materials evaluation is a continuing program.

### IV. RESPONSE TO ANALYTICAL REQUESTS

- A. Objective: To provide analytical support to R&D and Operations personnel and projects.

- B. Results:

Analyses and investigations by project personnel during the month of November included:

Several customer complaint samples were analyzed for possible contaminants.

Ten smoke condensate samples were quantitatively analyzed for catechol.

The amount of decanoic acid was determined on several samples of RL. These were pilot plant samples to which decanoic acid had been added at three different levels.

PROJECT NUMBER: 6906  
PROJECT TITLE: Biological Effects of Smoke  
PROJECT LEADER: J. M. Penn  
WRITTEN BY: W. R. McCoy and J. M. Penn  
PERIOD COVERED: November, 1988

#### I. INHIBITION OF EGF BINDING ASSAY

- A. **Objective:** Evaluate the role of catechol in CSC activity as measured by the EGF assay.
- B. **Results:** An experiment was performed to compare the effects of 2R1 CSC dissolved in acetone and methanol since the chromatographic removal of catechol from CSC would require the use one of these solvents. As compared to the standard DMSO solvent, both acetone and methanol produced the same magnitude of inhibition of EGF binding and reduction in cell numbers. Therefore, either solvent can be used without affecting CSC activity in the EGF assay.
- Two experiments were conducted to determine the binding kinetics of catechol as compared to 2R1 CSC. Binding analysis indicated that catechol and 2R1 CSC reduced receptor numbers as well as affinity.
- C. **Plans:** Conduct experiments using a broader range of EGF concentrations to distinguish between a change in affinity and the loss of high affinity receptor sites.
- D. **Reference:**

Patskan, G. Notebook No. 8738, p. 31.

#### II. SALMONELLA/MICROSOME (S/M) ASSAY -- CROSSED SOLUBLES/BASE WEB STUDY

- A. **Objective:** To determine if there is a concentration effect during the ultrafiltration process of BuS1 which has been retained and passed through a 1000 MW filtration membrane.
- B. **Results:** BuS1 was carried through the ultrafiltration process at various concentrations (0.1-0.5 mg/ml). The results showed that none of the test samples were significantly different from the BuS1 control. There was also no distinct concentration effect for either of the material that passed through or that which was retained.
- C. **Plans:** There are no immediate plans to continue work on ultrafiltration.
- D. **Reference:**

Thompson, L. H. Notebook No. 8731, pp. 1-14.

III. S/M ASSAY -- EVALUATION OF D-ALANINE

- A. **Objective:** Determine the activity of d-alanine as a pure compound in the S/M assay.
- B. **Results:** D-alanine was evaluated using the standard S/M protocol for pure compounds in the presence and absence of S9 in strains TA98 and TA100. The results indicated that there was no S/M activity of d-alanine under any test condition. In addition, there was no evidence of toxicity at any dose tested.
- C. **Plans:** Evaluate 5 and 10 mM of d-alanine oversprayed onto 1R4F using water as the solvent.
- D. **Reference:**

Thompson, L. H. Notebook No. 8731, pp. 15-20.

IV. PROTEIN KINASE C (PKC) IN INTACT CELLS

- A. **Objective:** To determine the dose response effects of CSC in the PKC assay.
- B. **Results:** Two types of CSCs (2R1 and CDL) were evaluated in the PKC assay. The results indicated that both CSCs produced qualitatively similar patterns of phosphorylation. However, there still appears to be difficulties with experimental variability. Several protocol modifications were implemented to improve reproducibility, but a significant amount of variability remains.
- C. **Plans:** Conduct experiments to determine if experimental variability can be eliminated.
- D. **Reference:**

Nixon, G. M. Notebook No. 8711, p. 46.

V. GLUTATHIONE DEPLETION ASSAY (GDA)

- A. **Objective:** To determine if CDNB or 2NF can be used as a control compound to examine the effects of 2R1 CSC induced reduction in GSH levels on TA98 mutagenic activity.
- B. **Results:** Two experiments were carried out to determine the effects of 2R1 CSC and CDNB pretreatment on GSH levels and TA98 activity of CDNB. 2R1 CSC at a dose of 0.2 mg/plate produced a 25% reduction in the GSH level, while CDNB at a dose of 50  $\mu$ M reduced the GSH level by 97%. Pretreatment of cells with 2R1 CSC or CDNB had no effect on the TA98 mutagenic activity of CDNB. A third experiment was conducted to determine the effects of 2R1 CSC and added GSH on the TA98 activity of 2NF and CDNB. Each tested in the presence of CSC (0.2 mg/plate) showed a reduction in activity (50% and 30% respectively). When each was tested in the

presence of CSC and added GSH (2.5 mM) a further reduction in activity (14% and 13%, respectively) was noted.

C. Plans: Determine the effects of added GSH and added NADP on the TA98 activity of 2NF and CDNB.

D. Reference:

McCoy, W. R. Notebook No. 8739, pp. 5-9.

PROJECT NUMBER: 6908  
PROJECT TITLE: Smoke Condensate Studies  
PROJECT LEADER: A. H. Warfield  
PERIOD COVERED: November, 1988.

I. TSNA PRECURSORS:

- A. Objective: To determine the precursors of MS TSNA.
- B. Results: A nicotine amino acid ester, *t*-butyl 2-(3-pyridinyl)-1-pyrrolidinylacetate (**1**), has been applied to a high nitrate, low alkaloid burley filler for the purpose of determining whether this material could serve as a model of bound nicotine. When the corresponding cigarettes were smoked, no significant differences in MS TSNA deliveries were found between these cigarettes and the control.

Work was continued to develop methods for determination of  $^{14}\text{C}$ -NNN and  $^{14}\text{C}$ -NNK. This methodology is needed to determine if MS  $^{14}\text{C}$ -TSNA formation occurs in cigarettes containing  $^{14}\text{C}$ -nicotine. Standard  $^{14}\text{C}$ -NNN,  $^{14}\text{C}$ -NNK and  $^{14}\text{C}$ -nicotine were successfully separated on an Ultrasphere ODS column using a MeOH/0.2% triethylamine phosphate (pH 7.2) gradient. A radioactivity detector was utilized.  $^{14}\text{C}$  activity between 0.1 and 0.5 nCi was detectable under these conditions. Also, work was carried out to expand previously developed TLC methodology to the isolation of an NNN/NNK fraction from 2R1 cigarette smoke which was spiked with labelled NNN and NNK.

An RL prepared from burley (Bu) CEL + sugars on Bu base web (BW) was smoked for MS TSNA deliveries. The sugars (glucose 7%, fructose 6.5%, and sucrose 0.9%) were added to mimic the sugar levels found in oriental (Or) CEL. When compared to a similar RL without added sugars, the TSNA levels found in MS smoke from BuCEL + sugars on BuBW were reduced by amounts similar to reductions obtained from 1/2 BuCEL + 1/2 OrCEL on BuBW as reported earlier. Reductions in NNN (60-62%) and NNK (52-53%) were almost identical for the two samples, while the sugar addition produced a 14% reduction in NNK as compared to a 38% reduction for the mixed solubles RL. When considered together with FTIR-EGA evidence, the data suggest that the reduction in TSNA is due to a reaction product (or products) formed from sugars and soluble ammonia.

- D. Plans: Since the *t*-butyl ester (**1**) did not result in TSNA formation the corresponding free acid will be prepared and the experiment repeated to determine if esterification may have prevented loss of the carboxyl group. When methods development is complete, cigarettes will be prepared from filler containing  $^{14}\text{C}$ -nicotine. The cigarettes will be smoked and the method used to determine if NNN and NNK are formed from nicotine during smoking. Also planned are sugar additions to other fillers, and overspraying of ascorbic acid on the sugar-treated fillers to determine if further reductions occur due to TSNA decomposition.

E. References:

- Haut, S. A. Notebook 8595, p. 188.  
Hassam, S. Notebook 8712, p. 62.  
Morgan, W. R. Notebook 8579, p. 96.

II. TSNA REMOVAL STUDIES:

- A. Objective: To explore the possibility of extracting TSNA from stored tobacco using an extraction fluid compatible with current processing.
- B. Results: Cigarettes were prepared from a control DBC burley and four DBC burley fillers which had been extracted with various mixtures of ethanol in hexane (EtOH/Hex) (using a cation exchange scrubber) in addition to the corresponding control. These cigarettes were smoked for TSNA delivery. The fillers varied considerably in nicotine and minor alkaloid content. The deliveries of NNN and NAT on a "grams filler consumed" basis correlated well with the concentrations of nornicotine and anatabine, respectively, but the delivery of NNK was not dependent on the nicotine content. The data were used to determine conditions needed for preparation of larger amounts of two extracted fillers. In one case, the objective was to prepare a Bu filler depleted in nicotine and TSNA but containing minor alkaloids, for use in future studies of TSNA pyrosynthesis. A 5% EtOH/Hex extraction was carried out to prepare this filler. In the other case the objective was to prepare a Bu filler with as low a TSNA delivery as possible. A 40% EtOH/Hex extraction was utilized for this objective. In both cases the cation exchange scrubber was used, and the extractions were run at room temperature. A separate extraction was carried out using resin that had been used for an earlier extraction and subjected to a cleaning process to determine if the resin can be recycled.
- C. Plans: The newly extracted fillers will be analyzed for TSNA and alkaloids, and MS smoke TSNA deliveries will be determined. The extracts have been concentrated, and their TSNA and alkaloid levels will be determined. If essentially all the TSNA and alkaloids were retained by the resin, the extracts will be added back to a portion of each filler. The low alkaloid/TSNA fillers, with and without extract addback, will be evaluated for subjectives.
- D. References:
- Haut, S. A. Notebook 8595, p. 188.  
Tickle, M. H. Notebook 8716, p. 94.

III. CROSSED SOLUBLES/BASE WEB STUDY (CHEMISTRY):

- A. Objective: To investigate the smoke chemistry of model cigarettes made from all possible combinations of solubles from bright, burley and oriental tobaccos on base webs from the three tobaccos.



- B. Results: Ten solutions representing fractions separated using an ultrafiltration membrane (nominal MW 1000) were sprayed onto bright base web (BrBW) and smoked. The CSCs were submitted to Project 6906 for Salmonella/microsome (S/M) assay.

The electrodialysis equipment to be used for removal of salts from CEL solubles has been obtained from the warehouse, and cleaned prior to being restored to service. A 240V service has been installed, and parts have been ordered.

Samples of the butanol and water fractions from a continuous BuOH/aqueous partition of BuSl were given to R. Izac for gel filtration chromatography. These two fractions as well as two peaks from gel filtration of BuSl will be submitted for spraying onto BrBW followed by determination of S/M activity of the corresponding MS CSC.

Solutions for 18 samples, including controls, have been prepared as part of the  $\text{Ca}^{++}$  add-on experiments and submitted (to R. Hellams) for spraying, and cigarette making. Subsequently, the CSCs will be submitted for S/M activity testing. Additional solutions representing various levels of  $\gamma$ -globulin added to BrCEL for spraying onto BrBW have also been prepared and await filler application, cigarette preparation, and S/M activity determination.

- C. Plans: No further work is planned involving ultrafiltration. A report will be written summarizing the  $\text{Ca}^{++}$  add-back data when all phases of the work have been completed. S. Hassam joined Project 6908 on November 1, 1988, and will begin assuming responsibility for the crossed-solubles base web studies.

D. References:

Magin, D. Notebook 8660, pp. 80-82.  
Hassam, S. Notebook 8712, p. 62.

IV. ANALYTICAL PROCEDURES:

- A. Objective: To develop and maintain analytical methodology for TSNA or other compound classes where information is needed for determining relationships of TSNA to their precursors.
- B. Results: Work continued on the development of a simultaneous VNA/TSNA analysis method. Precuts from the alumina column using methylene chloride and a mixture of hexane/methylene chloride were not successful in cleaning the VNA fraction of the components which cause the GC/TEA precolumn to deteriorate. Testing of a different precolumn packing is currently being performed.

The GC/NPD method for nicotine and minor alkaloids was reestablished. Initial problems with the collector on the HP 5880 GC were solved after the collector was replaced. The method was used

for analysis of 60 samples submitted by Project 1704, as well as several fillers from other studies carried out in Project 6908.

C. Plans: Continue development of the VNA/TSNA method.

D. References:

Morgan, W. R. Notebook 8579, p. 70.

Tickle, M. H. Notebook 8716, p. 94.

Levins, R. J. Notebook 8672, p. 183.

PROJECT NUMBER: 8101.  
PROJECT TITLE: Cigarette Testing Services Division  
SECTION LEADER: Jane Y. Lewis  
PERIOD COVERED: November, 1988

I. MARKET ACTIVITY

A. Objective: To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. Results:

1. Domestic

Brown & Williamson is importing Prince 85 and Prince Lights 85 cigarettes from Denmark. These cigarettes are manufactured by Scandinavian Tobacco International and are being sold in Arkansas. These cigarettes are targeted toward the young adult male smoker. The Prince cigarette delivers 16 mg tar and the Prince Lights cigarette delivers 11 mg tar.

The Liggett Group is test marketing Chesterfield Lights 85 and 100 and Savvy Lights 100 (plain and menthol) and Savvy Ultra Lights 100 cigarettes in St. Louis, Missouri and Peoria, Illinois. This standard priced brand has a \$3.50 coupon that is part of the carton. No tax stamps were on the cigarette packs. The Chesterfield Lights cigarettes deliver 11 mg tar; the Savvy Lights cigarettes deliver 10 mg tar and the Savvy Ultra Lights cigarettes deliver 6 mg tar.

2. International

R. J. Reynolds has introduced Salem Lights King Size (Box) cigarettes in Japan. This brand delivers 8 mg tar, 0.5 mg nicotine and 0.2 mg smoke menthol and is similar in most characteristics to Salem Lights King Size (Soft Pack) currently on the market in Japan.

American Tobacco is exporting Carlton Plus King Size cigarettes to Japan. This brand delivers 6 mg tar and 0.5 mg nicotine.

Brown & Williamson has introduced Capri 100 Box (plain and menthol) cigarettes in Japan. These cigarettes have a 17 mm circumference and the blend contains no expanded stems or expanded tobacco. The plain cigarette uses a different type of reconstituted material than the menthol cigarette. The reconstituted material in the plain cigarette uses softwood fibers, tobacco leaf and stem; the reconstituted material in the menthol cigarette contains no softwood. Capri 100 has a dual carbon in CA/CA filter and delivers 8 mg tar; Capri Menthol 100 has a single CA filter and delivers 10 mg tar.

## II. ANALYTICAL METHODS DEVELOPMENT AND SUPPORT

A. Objective: To evaluate and recommend analytical methods and new technology in support of programs for R&D and Manufacturing.

B. Results:

1. Monitor

A low delivery smoking monitor (LDM#6), to be used by P.M. USA, P.M. International and the Gulf Coast Countries, was fabricated in Semiworks on October 24, 1988. These cigarettes were equilibrated for 11 days at 60% R.H. and 75°F. Two hundred and eighty-two cases were canned and these have been shipped to Southern Cold Storage. Calibration of this monitor will begin in January, 1989.

2. Firmness

Diagnostic evaluations of the FTR cigarette firmness instrument are being conducted by Chuck Higgins of Development Engineering. Electronic malfunctions have been attributed to interferences caused by environmental vibrations. A modification to the electronic board has been made to shield the signal. Comparative studies are being conducted to determine the influence of this modification.